CHNOLOGY DEPT.

THE Chemical Age

VOL LXX 9 JANUARY 1954 No. 1800

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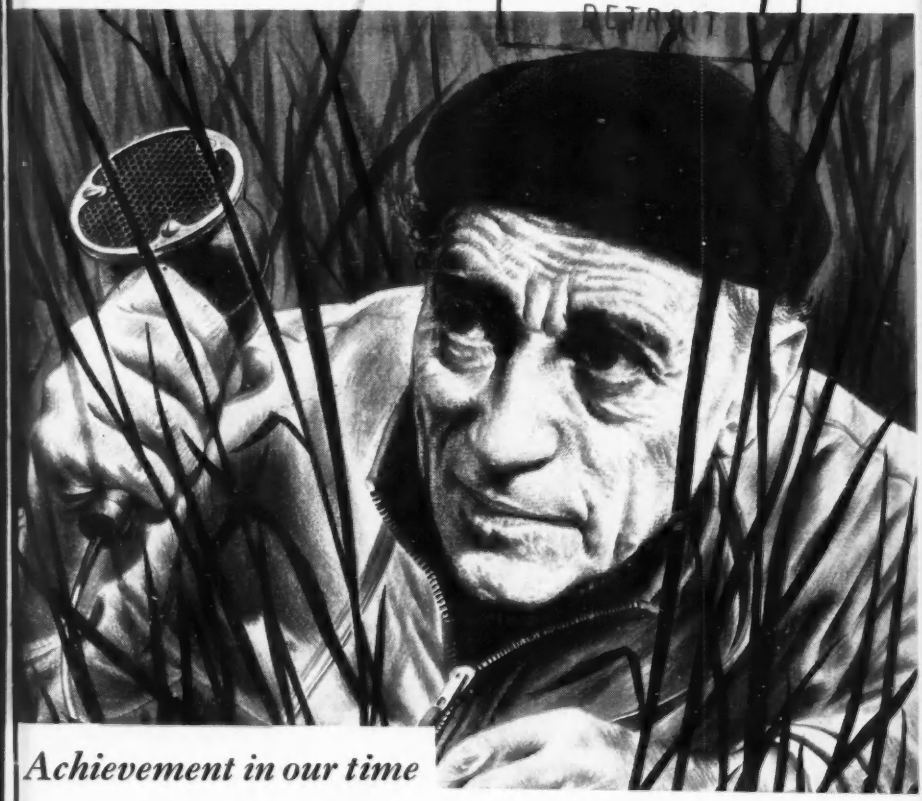

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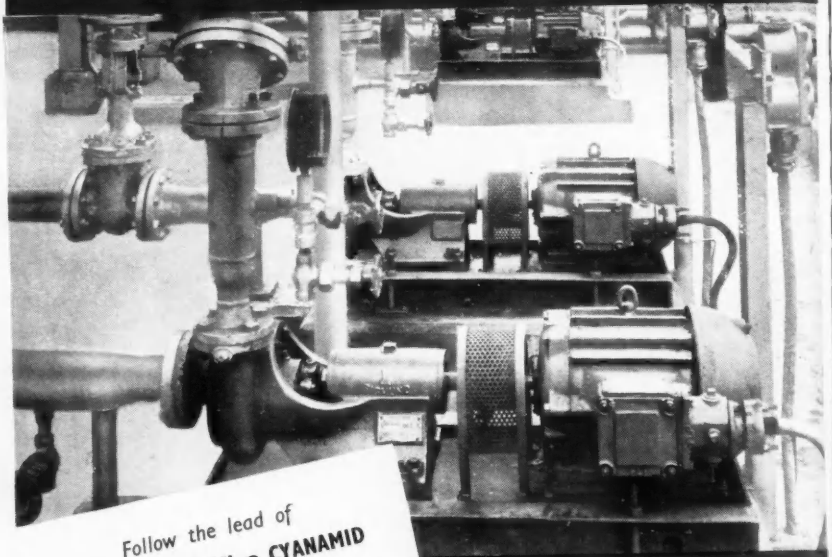

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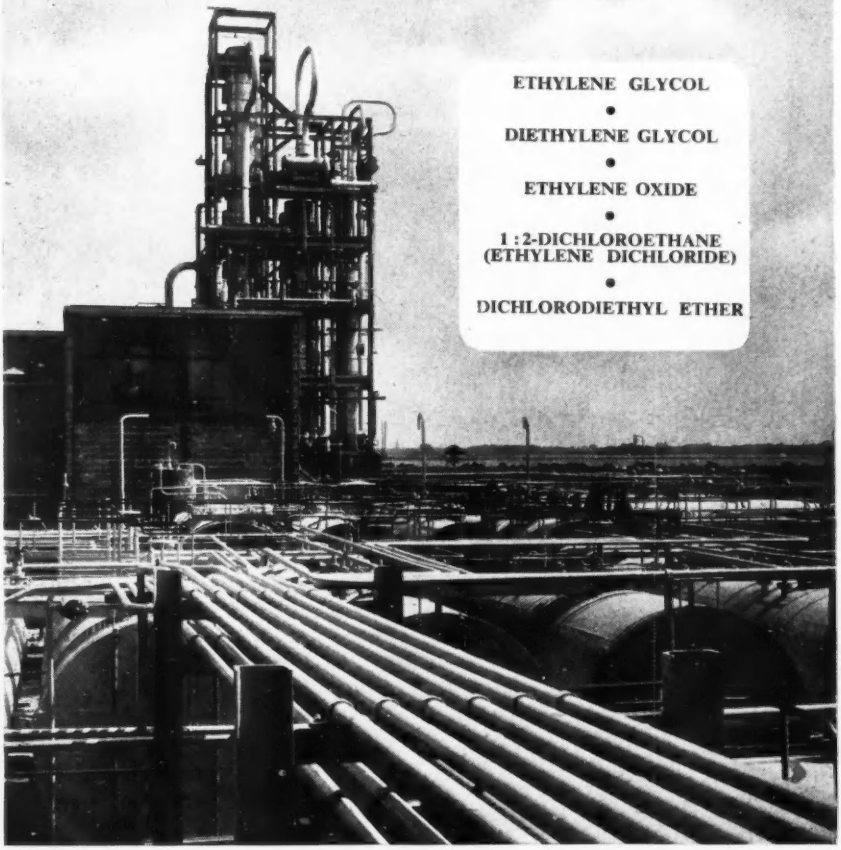

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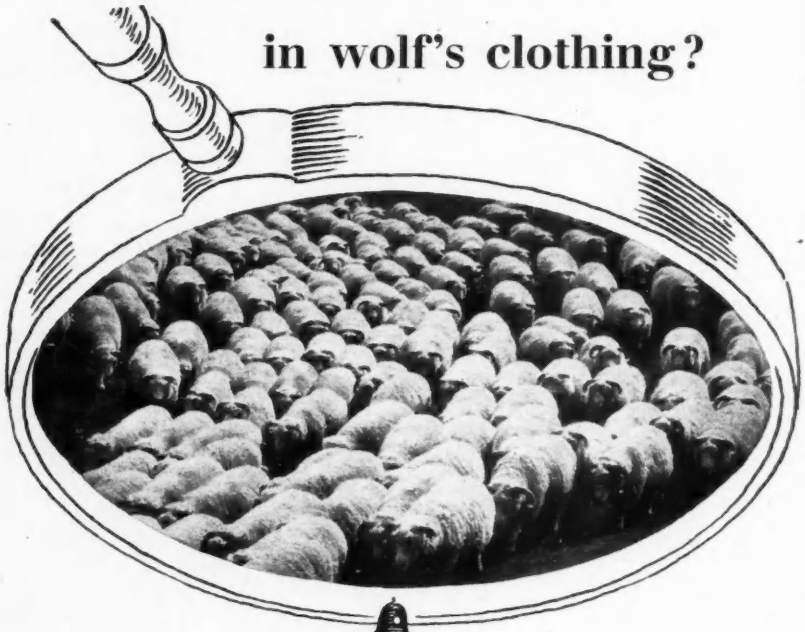

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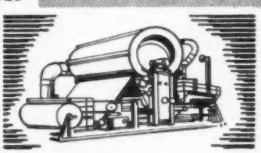

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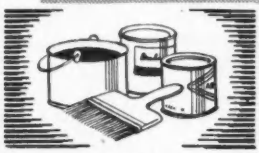

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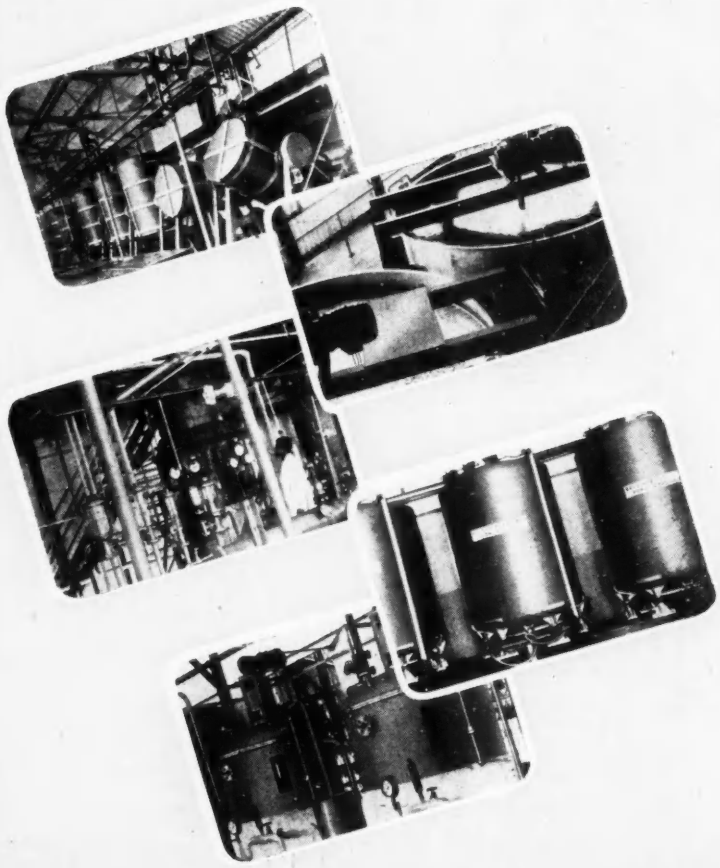

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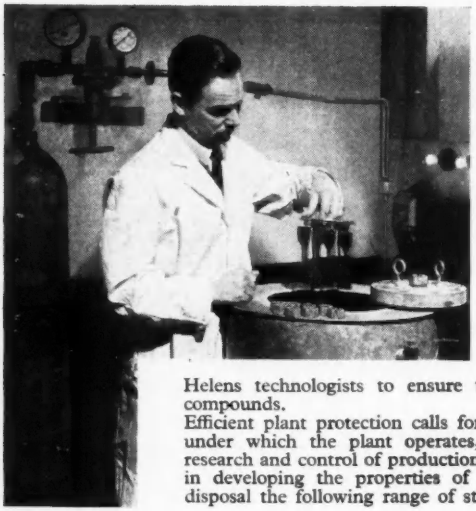

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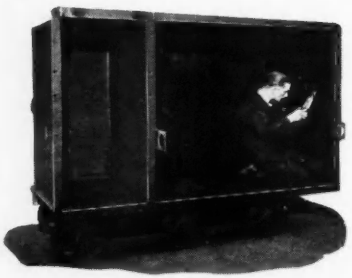

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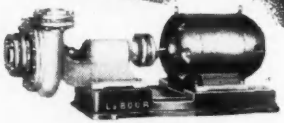

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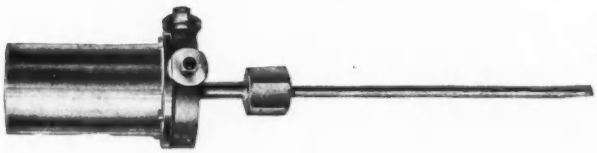

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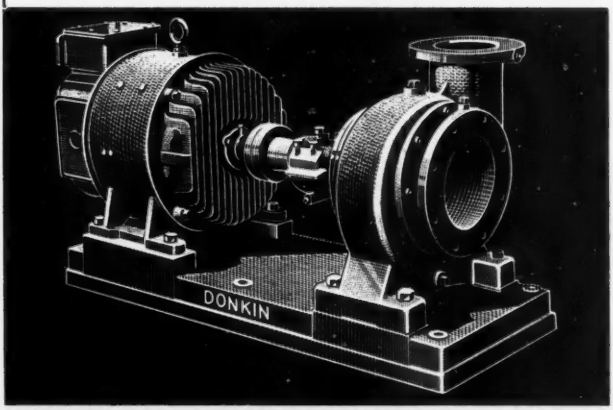

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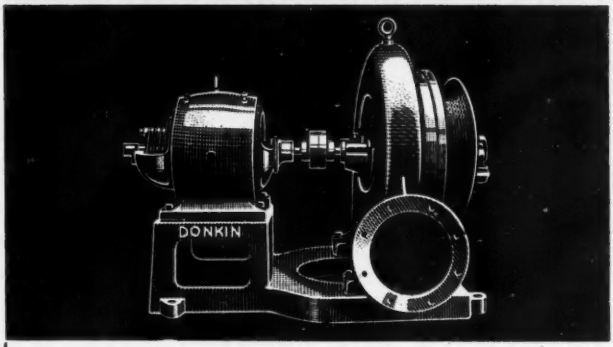

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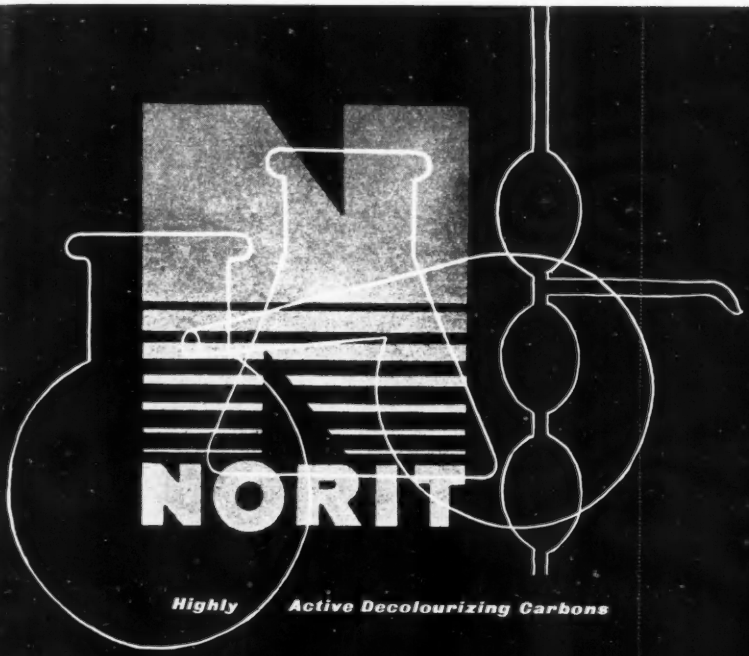

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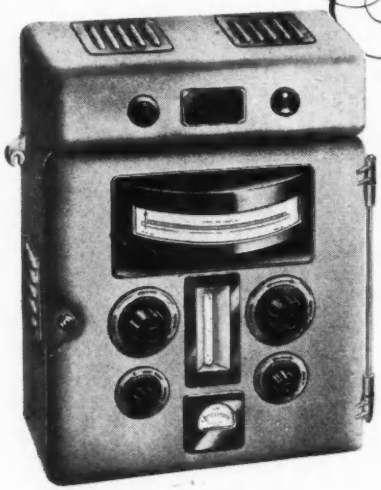

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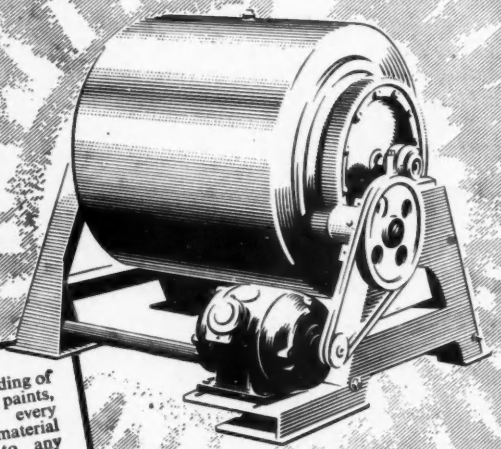

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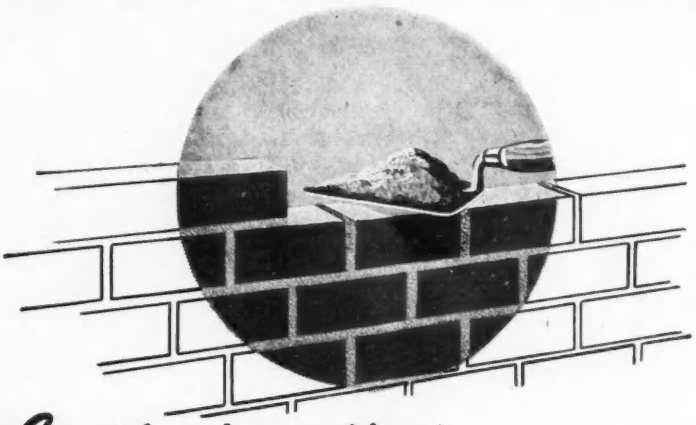

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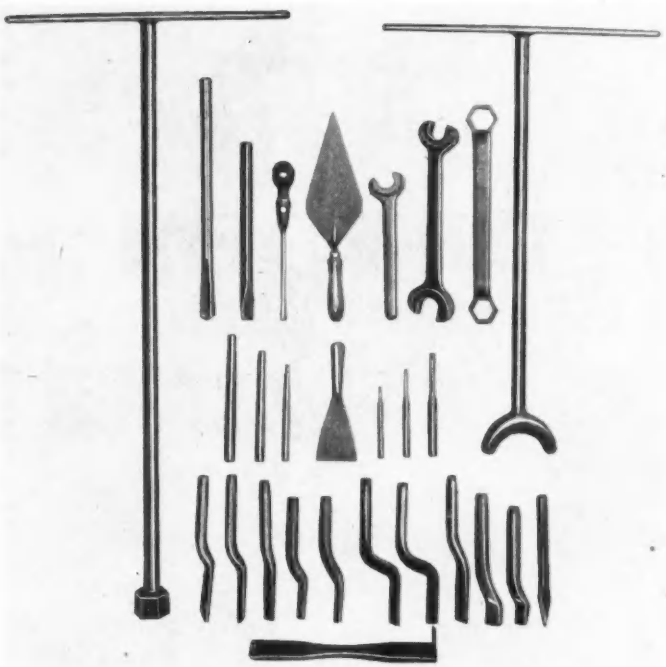

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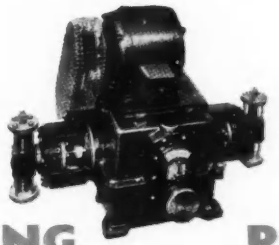

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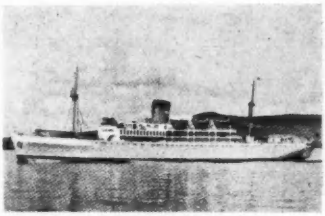

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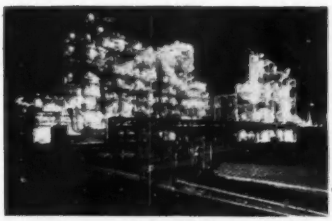

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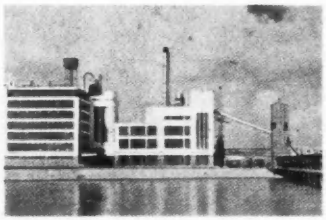

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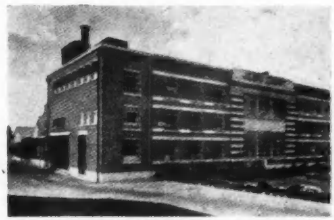

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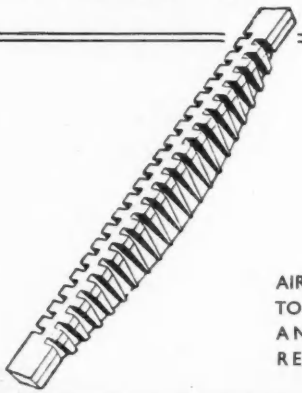

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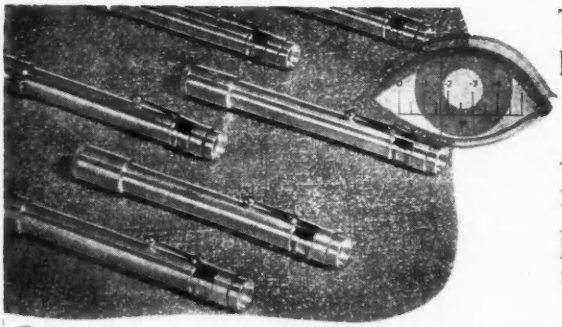

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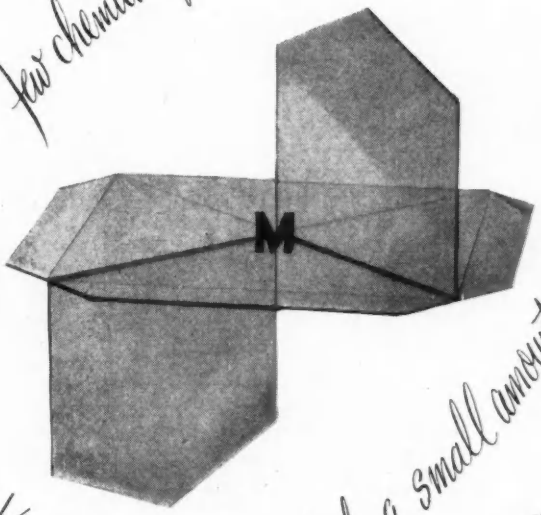

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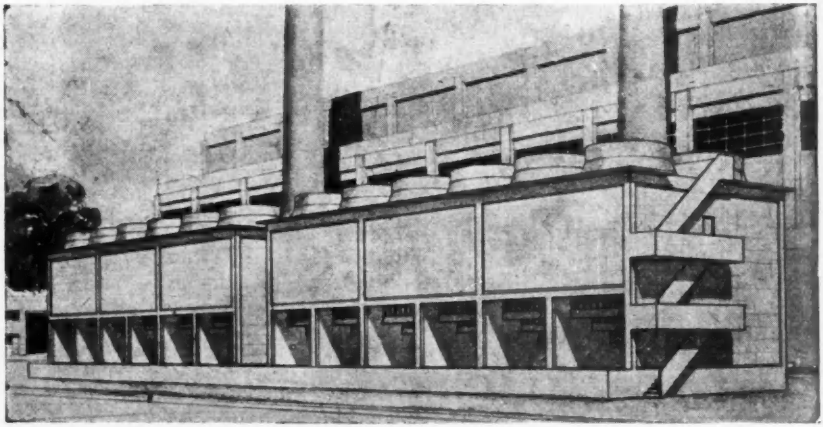

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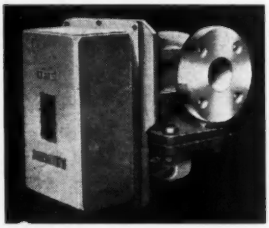

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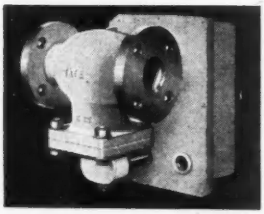

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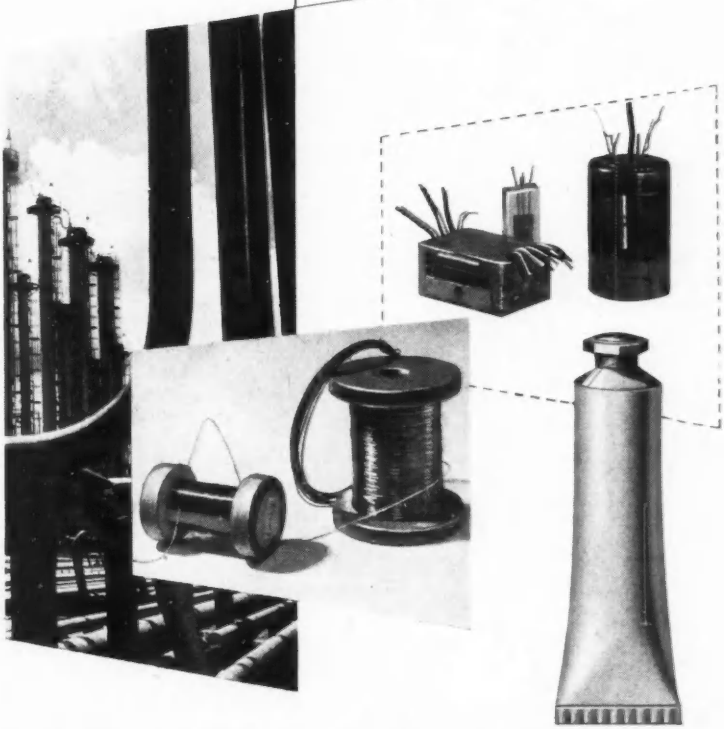

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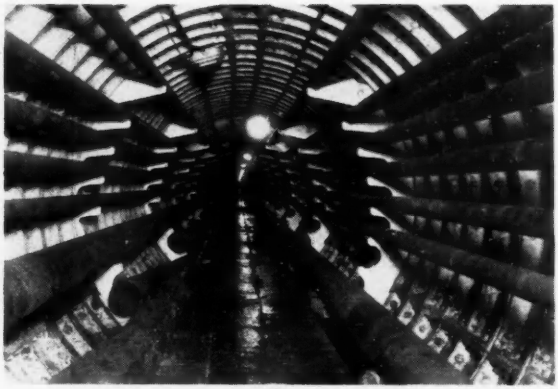

Photograph by courtesy of Imperial Chemical Industries, Ltd.

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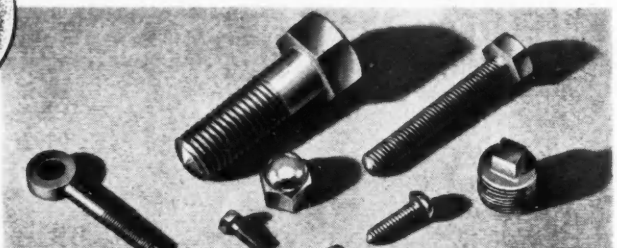

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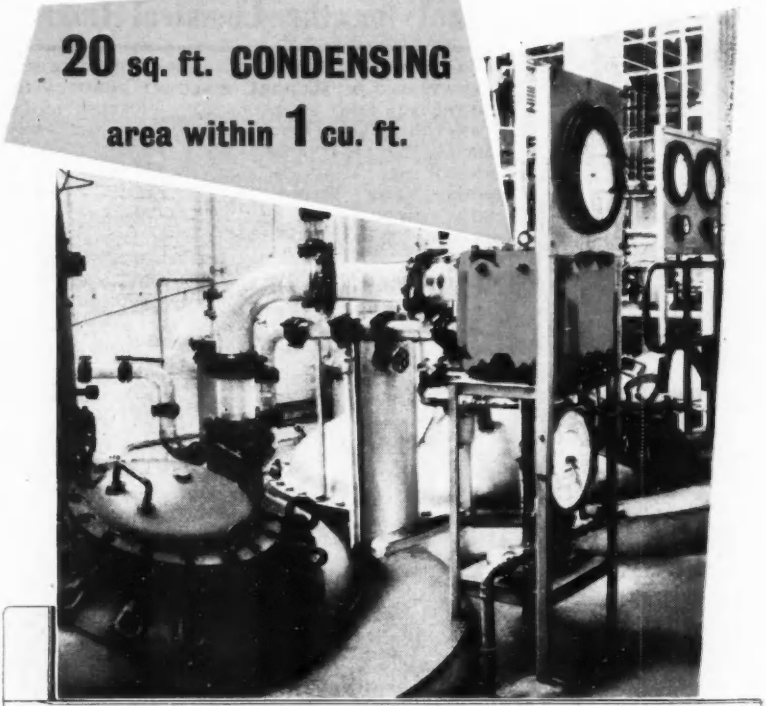

A typical Condenser installation—Photograph by kind permission of British Drug Houses Limited.

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CONDENSING FLUID	Thionyl Chloride & Hydrochloric acid & Sulphur Dioxide.
COOLING FLUID	Brine at 32 F.
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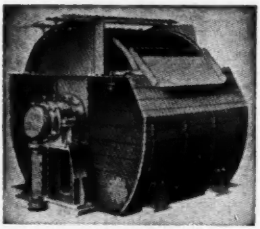

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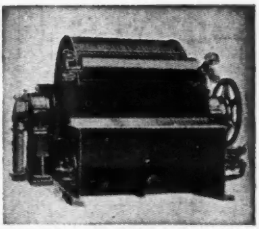

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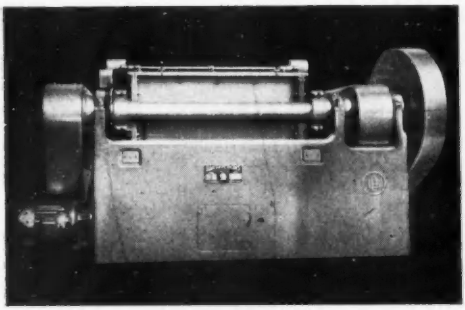
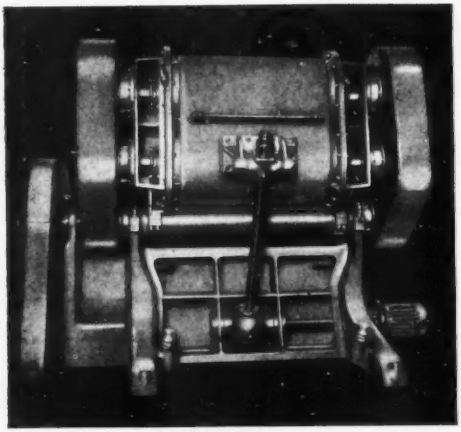


Above. Front view of a Size 17 Type VIII Class B.B. for 176 gallons per mix.

Below. Rear view of same machine tilted for emptying.



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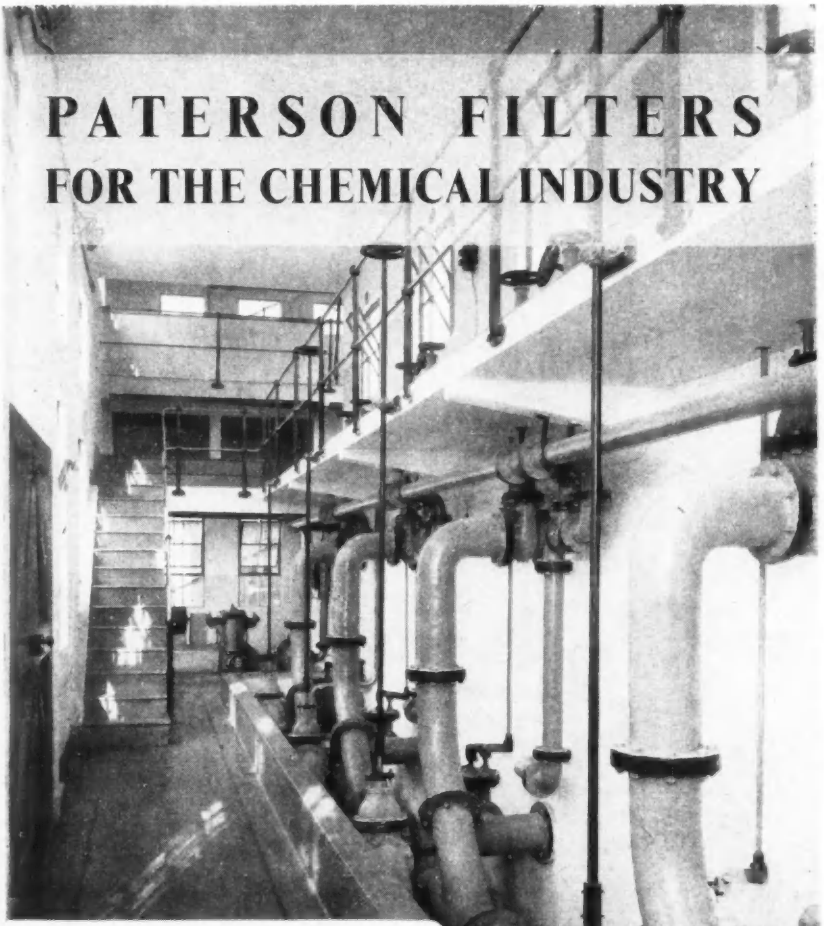

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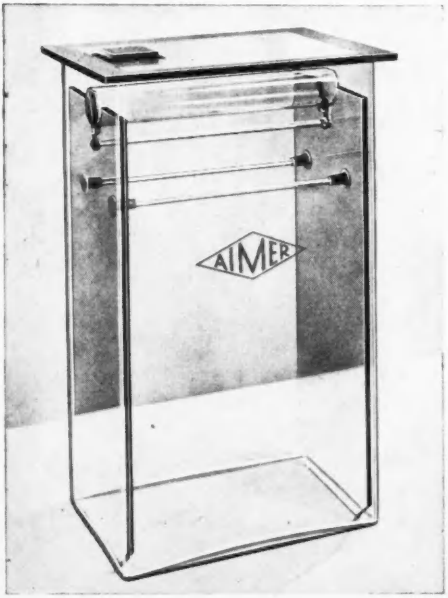

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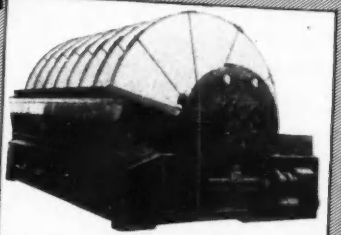

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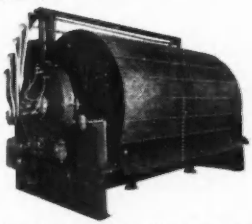

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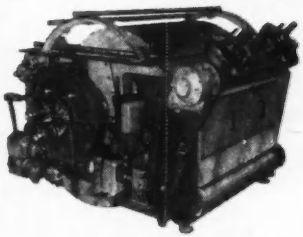

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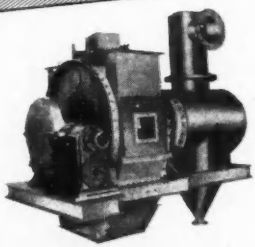

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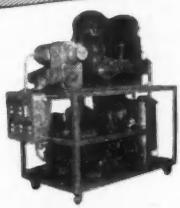

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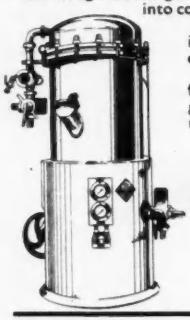

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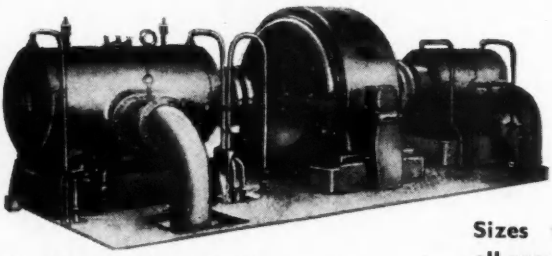

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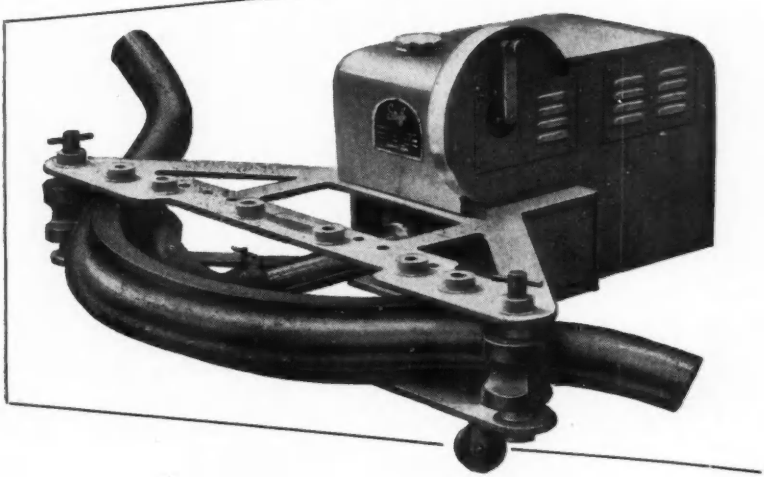

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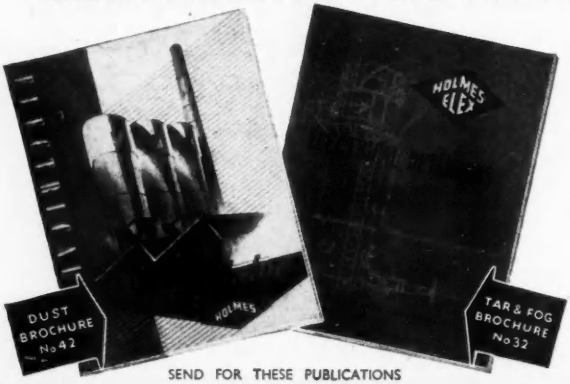

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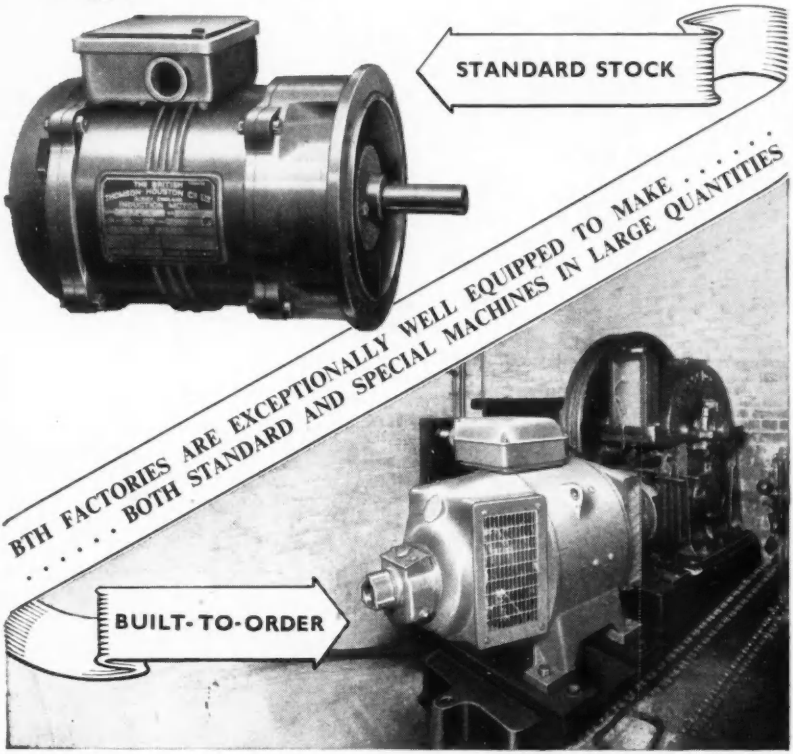
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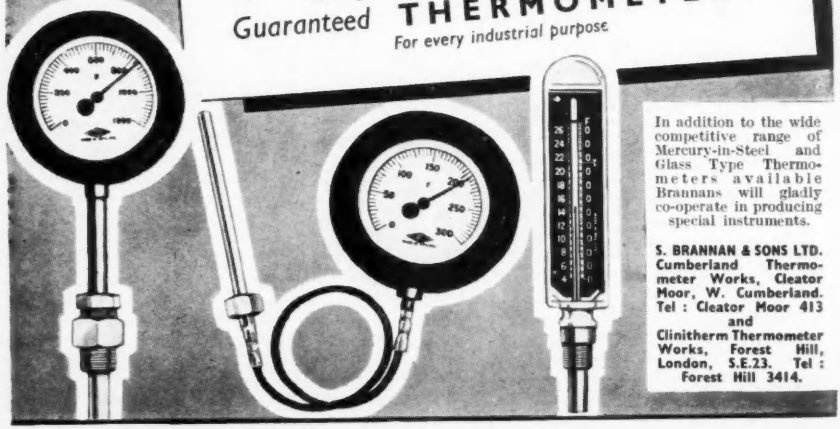

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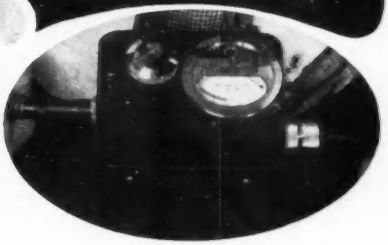

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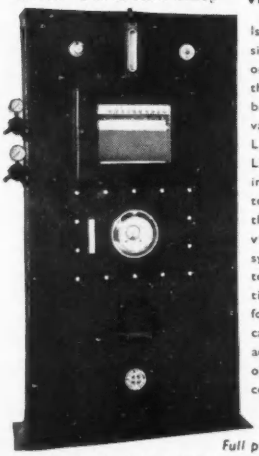

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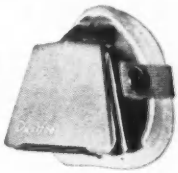

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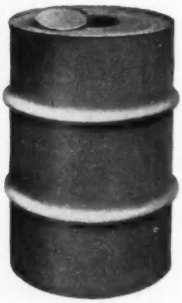

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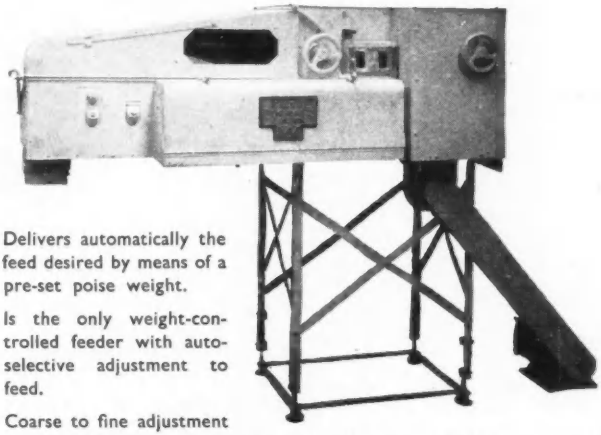

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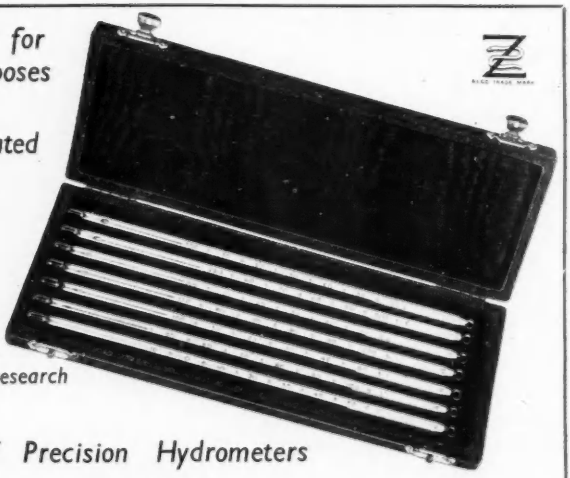

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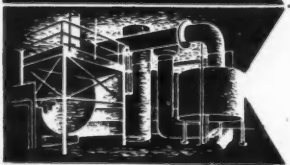

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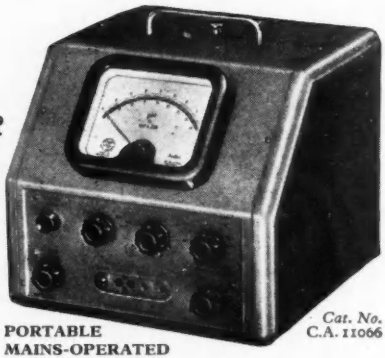

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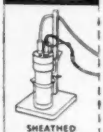

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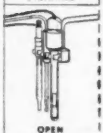

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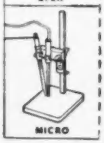

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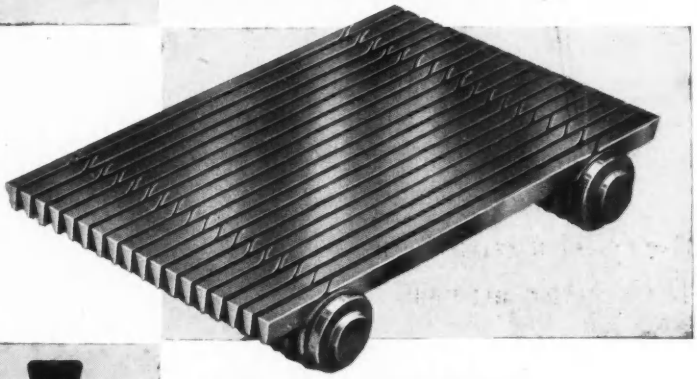

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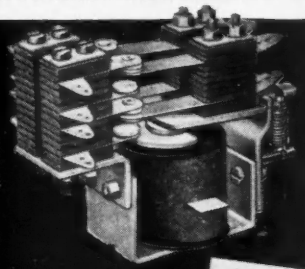

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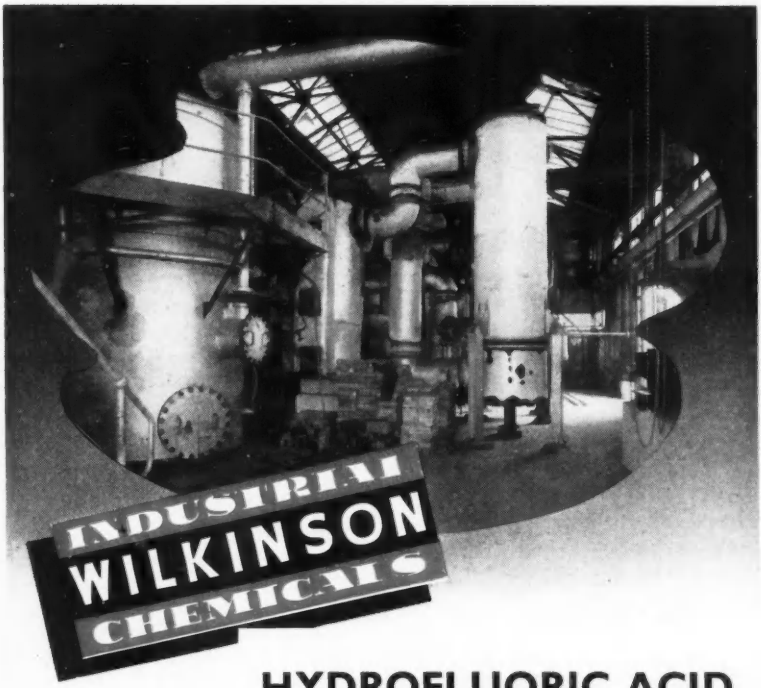

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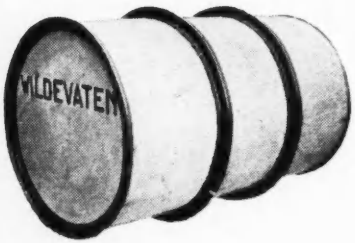

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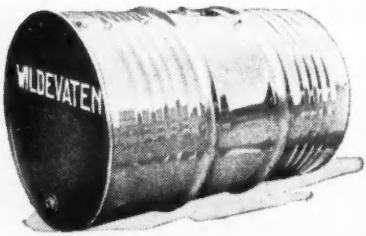

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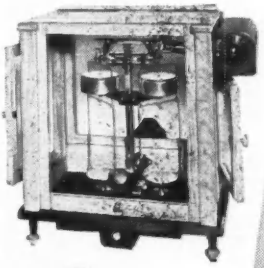

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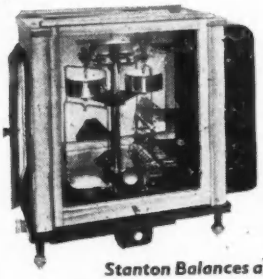

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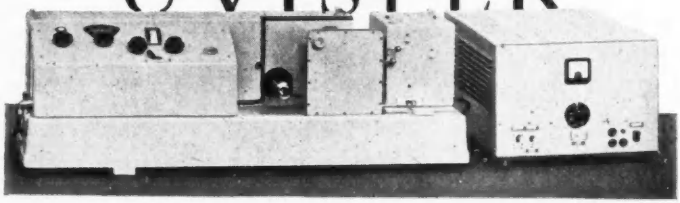

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1954

WHEN last year we attempted to forecast the prospects of 1953, we said in looking back that 'the wicket could have become much stickier in 1952 but the expected revival of German competition did not manifest itself at all strongly', and 'it may well be that 1953 will prove the first post-war year of realistic challenge'. Now, at the onset of 1954, we cannot once more look back upon a doubtful resurgence of German chemical production. The age-old European competition for chemical trade with the rest of the world has returned. Western Germany's total exports of chemicals in only the first five months of 1953 rose by 26 per cent and her exports to the United States showed the sharpest rise of all. This excludes fertilisers; if these are brought into the balance sheet the advance of German export trade has been greater still. In the first nine months of 1953, Western Germany sold over $6,000,000 worth of nitrogenous fertilisers. In 1954 the German chemical industry is likely, in the words of a contemporary US journal, 'to make an all-out bid for its former export supremacy' (*Chemical Week*, 1953, **73**, [24], 14).

It might at first sight seem disconcerting that Germany has been able to expand her dollar exports so decisively at a time when we seem unable to achieve similar and badly-needed expansion. Part, though certainly not all, of the explanation is to be found in the political scene. The United States has a stake in Western Germany and the call of 'trade, not aid' echoes in Washington and New York with somewhat sweeter force. If there is in fact no trade agreement treaty with a 'favoured-nation' clause, there is for the time being an American determination to make sure that Western Germany recovers her economic strength. It requires only the briefest appraisal of the present international situation to see that this is likely to continue. It is the other part of the explanation that is more profitable and practical for British manufacturers to study. The German approach to export selling is more aggressive. According to American accounts, a network of agencies is being vigorously re-established in the United States. German goods are not merely 'offered', they are sold. One of our principal tasks for 1954 must be to remove every discernible weakness from our export selling technique.

The recurring dollar crises of our own post-war recovery have perhaps over-emphasised the US market for exports. Too often we have had to refuse trade with non-dollar countries so that what goods we could spare could be exchanged for US currency. Assuming that our much safer sterling/dollar equilibrium can be maintained, 1954 will be well used if we can recover some of the non-dollar export trade that we were circumstantially forced to discard or ration. The world is big and its ever-rising population demands more and more chemical supplies, nor will true recovery of Britain and the sterling area come until every currency is welcome credit in London. As for dollars, we might well remember that for some time now the Canadian dollar has been worth fractionally more than the US dollar. Can the lost post-war opportunities of investing in Canada's rapidly rising chemical industry be compensated by seizing the opportunities that remain? Canada's non-primary chemical industry is still only beginning to emerge. It is not too late for British companies of experience

to take the lead in establishing Canadian subsidiaries. A notable example was set in 1953 by Watford Chemicals Ltd., who have 'parented' a Watford (Canada) offspring in Toronto to manufacture esters of the fatty acids and polyhydric alcohols. Canadian comment is worth reproduction: 'In entering into the ester field in Canada, Watford Chemicals have made an entrée into one of the most promising fields . . . for the speciality chemical manufacturer. Consumption of this line of chemicals has risen spectacularly in the last decade, and the curve is still turned upwards. . . .' (*Canadian Chemical Processing*, 1953, **37**, November, 76.)

What technical developments at home are likely to assume major importance in 1954? This hardy annual task of the leader writer is not unlike predicting the winner of the Derby. In the metallurgical field, the surest 'tip' is titanium. Although a British titanium metal plant is now being erected, it will not commence production until the end of 1955; in the last few months, however, the home requirements for titanium in the USA have risen very sharply, and there are solid fears that significant titanium exports from that country will vanish by the middle of 1954. An 18-months period of titanium scarcity may be a crippling blow to British aircraft and turbine engine development, a blow to our competitive position if not to current production. The I.C.I. pilot plant that is already producing some titanium metal here will at best produce only about 150 tons a year, and even this rate of output has still to be attained. *The Economist* (1953, **169**, 905) recently raised the question whether the British project needs to be enlarged and accelerated. It is at any rate a question for the Government and the Ministry of Supply, for both in America and Britain titanium must remain a heavily subsidised element unless some cheaper means of extraction than the Kroll process can be developed.

A development of long-term importance for this country is that of mixed fibres. The natural and synthetic fibres each have their special properties, but the time has now arrived when combinations of fibres must be more actively considered. Blending cotton with synthetic fibres, and getting well ahead in this new field, could easily be Lancashire's salvation; for if German chemicals are back in force, so, too, is Japan's cotton, and British exports of textiles must be inimitably superior if this traditional overseas trade is not to dwindle and decay. The Shirley Institute is beginning a new programme of research on spinning, weaving, and finishing mixed fibres of cotton and nylon. This is a field in which American technology already holds a lead. We might not be starting from so rearward a base if synthetic fibres had not been looked upon as enemies of natural fibres and the natural fibre textile industry.

As in previous years since the war, oil refining and the production of chemicals from oil will retain a forefront position in the march of British technology. Within only five years, the UK refining capacity had been raised from 2,500,000 tons to 25,000,000 tons per annum. Fawley, Grangemouth, Shellhaven, Stanlow, and the Isle of Grain refineries may well be regarded as our greatest post-war achievements. Even the smallest of these new plants can refine as much oil as the whole British industry could refine in 1947. The full currency-saving effect of these refineries has only just begun to make itself felt and, in addition, the export of refined products is bringing in substantially over £50,000,000 a year. Subsidiary in a sense, yet in a sense intermingled from the start, is the petrochemical industry. In 1954 we may well see the petrochemical industry assume its full stature.

We may be facing a more competitive year. A changing international background may bring some reduction in the demands of defence, a continuation, indeed, of the process that began in 1953 with the Korean truce talks. Orders may be harder to get, and in the Western world as a whole new private demands may not replace at once or in full the slackened demands of state departments. But there is no evidence that major recession or depression is 'round the corner' and where manufacture is soundly backed by up-to-date research and service there is no reason to fear that trade will be scarce. Our fate, as a chemical trading nation, is in our own hands.

Notes & Comments

The Big & the Little

'AS a general rule, workers go absent for one reason or another proportionally more frequently in large concerns than they do in small concerns.' This is the conclusion reached in a study of absenteeism by the Acton Society Trust, and it has been based upon comparisons of actual absenteeism rates in large coal mines, a large industrial organisation with 91 separate units, and a chain of retail stores, with average rates of absenteeism. A correlation between large unit size and higher absenteeism was markedly found in coal mining, fairly marked in the large industrial organisation, but 'only just discernible' for the chain stores. It is perhaps too easy to draw the further conclusion that personnel management is more efficient, or at any rate more effective, in small units of employment. The greater intimacy of personal relationship possible in the small works with its 'on-the-spot' head office should, in theory at any rate, stimulate the highest possible standards of conscientiousness. In practice, however, it is possible for this natural advantage to be lost by bad management; the worker's sense of loyalty can be readily acidified if close contact reveals, in fact, or only apparently, too many examples of managerial incompetence and indecision.

Personal Attention Important

ONE might expect the average effect of 'small unit size' upon the rate of absenteeism to be small, neither positive nor negative, with some small firms gaining greatly through their close control and others losing. If this is so, then the conclusion of the Acton Trust study indicates that 'large unit size' brings its own negative effect, that absenteeism rates for smaller organisations represent the normal and that the higher rates of large organisations represent something solely attributable to the disadvantages of remote final management. The increasing efforts of large concerns to create a stronger corporate and personal atmosphere throughout their staff organisations would certainly seem in the right direction. After all, it is humanly understandable. Whatever our status for the time being, whether as employee or consumer, we dislike the feeling that a decision about our affairs is ultimately taken by some unknown person in a far-away office. Personal attention based upon full knowledge of the circumstances is still one of the most satisfying features of everyday life.

Surface-Activity & Fertilisers

'THE original enthusiasm generated for surfactants or wetting agents in processing phosphate rock and fertiliser mixing seems to have subsided somewhat since a year ago.' So began a short review of the development in a recent issue of *Agricultural & Food Chemistry* (1953, **1**, 1044). A fairly familiar pattern in American technological novelty appears to have been followed—great initial optimism unpreceded by thorough tests, then the slow harvest of doubts from a crop of uncertainties and inconsistencies. There were three benefits claimed; shortening of the time required for curing acidulated rock, quicker ammoniation of superphosphate, and reduced caking with compounds. In each of these the *modus operandi* was theoretically the same, the reduction of surface tension in the liquid phase of the mixed materials and possibly also some reduction of tension in the solid phase. Of the three, the acidulation of phosphate rock seems to have been the most hopeful field for surface-active agents. Here there have been fewer inconsistencies of performance. It is said that one of the principal difficulties is to know which agent to select. This in itself reduces the strength of the theoretical argument for if the principle is sound numerous surfactants should be able to demonstrate benefit.

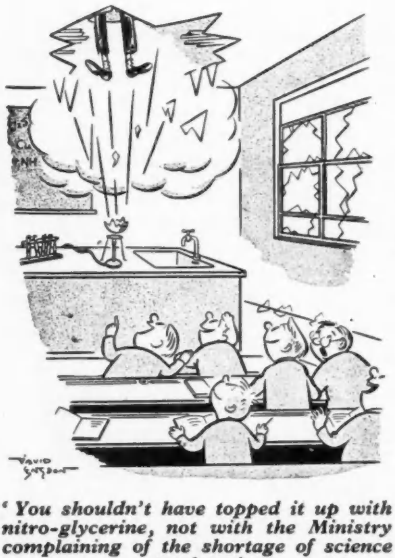
Inconclusive Stage

ALMOST inconclusive stage has been reached after little more than a year of empirical, guess-and-test

efforts in the factories, and the US fertiliser industry is on the whole happy to pass the entire subject over to more fundamental research investigations at the USDA centre at Beltsville. A promising idea, but possibly one from which too much has been expected, has had a delicate infancy; its further development, if any is to be made, will have to be much slower.

Must Not Dismiss Lightly

IT has been suggested that in any case the use of surface-active chemicals will find little scope in the British industry. It is true that ammoniation is at present only practised on a small scale, and the caking problems of compounding are largely overcome by granulation. But acidulation of phosphate rock is carried out very widely, and there is also the possibility that the process of wetting mixed fertiliser to prepare the granules could be run on a reduced quantity of added water in the presence of suitable surfactants. In theory at least these possibilities should not be swiftly dismissed; they may exist in actuality but not perhaps with a sufficient degree of improvement to justify the cost and trouble of a trace-adding operation.



'You shouldn't have topped it up with nitro-glycerine, not with the Ministry complaining of the shortage of science teachers'

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In America, too

THE shortage of science teachers in schools is not confined to the United Kingdom (see THE CHEMICAL AGE, 1953, **69**, 1313). A new report, based on an educational conference held at Harvard, has warned the United States that there is a current shortage there and it is likely to become more severe in the future. Some teachers now responsible for science in US high schools are inadequately qualified. By 1960 there will be an increase of 41 per cent (or, in real figures, of 2,700,000) in public school students; by 1965 there will be a further increase of about 2,000,000. This corresponds with a 1960 requirement of 84,000 science teachers and a 1965 requirement of 100,000; the present figure is 67,000. At the same time the output of college graduates is sharply dropping. In 1950 it was 430,000; in 1953, about 300,000. The output of graduates trained for high school science teaching has dropped in the same brief period from 9,096 per year to 4,665. These discrepancies between rising demand and falling supply seem even worse than those to be faced in Britain. No reason is stated for the severe falling-off in graduate output, but we may not be far from the mark in surmising that one highly influential factor is the absence in America of any clear-cut plan for easing or deferring the university students' military obligations; at any rate, this topic has been frequently ventilated in US scientific journals and news publications. It is almost comforting to learn that the United States has no immunity from this grave post-war problem. It will, however, be interesting to see whether remedial actions taken there are on a bolder scale and imbued with a greater sense of urgency than actions to be taken here.

New Year Messages . . .

Professor C. K. Ingold, F.R.S., D.Sc., Ph.D., A.R.C.S., F.I.C.

(President, The Chemical Society)

THIS message of good wishes and best hopes for the prosperity and success to all chemists in 1954 is based on the tolerably sound principle of the short extrapolation. In the year 1953 seeds have been sown, probably more than can yet be recognised for what they are, which will determine future developments in pure chemistry, and some of them in the applied science.

Earlier seeds have reached a stage of growth at which the shape of things to come can be seen, as has been described, to give a single example, for the borderland of chemistry and biology, in two remarkable lectures delivered in 1953 before the Chemical Society. Biological chemistry is a form of high-polymer chemistry with which the ordinary methods of organic chemistry cannot cope, and Professor Tiselius, in his Centenary Lecture, described a whole range of special physico-chemical tools for the separation and characterisation of the individuals composing natural protein substances.

And as to growth, the mystery of self-synthesis seems at least to have been lessened. Carrying on from the template idea, which Pauling did so much to establish, Sir Cyril Hinshelwood, in his Faraday Lecture, showed how the ordinary principles of chemical kinetics could be used to construct a model which reproduced the appearance of self-synthesis in a system of substances, as well as variations on normal growth, with death as a particular case, similar to those observed by the suitable treatment of unicellular organisms. In a very different field of high polymer chemistry, the arrangements now being made for the much greater production and distribution in this country of essential monomers show that the plastics industry in Britain can look forward to a period of rapid progress.

* * *

Sir William Ogg, M.A., B.Sc., Ph.D., LL.D., F.R.S.E.

(President, The Society of Chemical Industry)

IT is one of the most pleasant tasks of any president to send good wishes at this time of year. As one closely connected with, though not directly engaged in, chemical industry, I have, as president of the Society of Chemical Industry, found a remarkable friendliness in that vast field at once so varied and so specialised which is now included in industrial chemistry, and I gladly avail myself of this opportunity to wish all those engaged in it a very prosperous year in 1954.

The times in which we live are hard, and with the renewal of foreign competition we shall have to make even greater efforts to maintain and increase the essential exports by which we in these islands live.

Not only does the industry face difficult times, but the scientific societies also have their problems. Costs continue to rise and obligations to members do not become less —I need mention only the recent decision of my Society to provide its members with abstracts of the literature on applied chemistry from the beginning of 1954 — and I would remind the individual chemist that the societies need his support.

Let us go forward in 1954 in that spirit of co-operation which has already given such fruitful results.

. . . From Trade and Professional Leaders

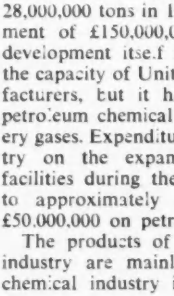
New Year Messages . . .

H. S. Gibson, C.B.E., M.A., M.I.Mech.E., F.Inst.Pet.

(President, The Institute of Petroleum)

SINCE the war a great petroleum refining industry has been built up in the United Kingdom where refinery capacity has increased from 2,500,000 tons in 1947 to 28,000,000 tons in 1953, involving the investment of £150,000,000. Not only has this development itself made great demands on the capacity of United Kingdom plant manufacturers, but it has also given rise to a petroleum chemical industry based on refinery gases. Expenditure by the chemical industry on the expansion of its production facilities during the same period amounted to approximately £250,000,000, including £50,000,000 on petroleum chemical projects.

The products of the petroleum chemical industry are mainly intermediates for the chemical industry itself. In 1952 United Kingdom production was estimated at 250,000 tons which has, to a large extent, replaced intermediaries imported from abroad. Thus not only has petroleum replaced coal as a major British export—in 1953 British exports of refined petroleum amounted to over £70,000,000—but in the chemical field the petroleum industry is also making a great contribution to the United Kingdom balance of payments by reducing the volume of imports from the dollar sources.



In the past the oil industry has drawn on the chemical industry for field and refinery technologists, but has repaid its debt in full measure by its contributions to the development of large-scale continuous process plant. The recent developments in the field of petroleum chemicals have drawn the industries closer together, as is illustrated by the joint meeting of the Society of Chemical Industry and the Institute of Petroleum in March, 1953, at which a symposium was presented on the production and utilisation of petroleum raw materials in the United Kingdom chemical industry.

The petroleum industry looks forward to still closer co-operation with the chemical industry and extends to its colleagues in that industry its best wishes for a prosperous year in 1954.

* * *

W. J. Worboys, B.Sc., D.Phil.

(Chairman, Association of British Chemical Manufacturers)

IT is my pleasure and privilege, as chairman of the Association of British Chemical Manufacturers, to extend New Year greetings to all our friends, both within and outside the chemical industry.

For our industry, 1953 has been a better year than 1952. Throughout the year there has been a steady climbing-up from the trough of the 1952 recession, and in the closing months of the year production in many sections of the chemical industry was at, or near, record levels. Home sales have reflected the general rise in industrial activity in the United Kingdom, and export sales, though slightly down on 1952, can be regarded as fairly satisfactory, if allowance is made for the continuing import restrictions in some important markets and for increasing competition.

During 1953 the chemical industry added over one hundred new products to its range and there was considerable capital expenditure on new and improved production facilities. The industry's expenditure on research work, directed towards improving existing processes and products, as well as towards discovering new products, continued at a high level. Good progress has been made by many firms in applying the principles of work study, and to further this work the Association of British Chemical Manufacturers organised a very successful conference at Buxton in October.

Forecasting is always difficult and dangerous, but the chemical field is a wide one, and the exploration of parts of it is only now beginning. In other words, the expansion potential of the chemical industry is great. This, coupled with the

fact that our industry can boast of good workers and of enterprising managements, means that we can face 1954 and the years ahead with hope and confidence.

I should like, on behalf of the chemical industry, to wish you all success and prosperity in 1954.

* * *

James Gray, M.B.E., T.D.

(President, Fertiliser Manufacturers' Association)

AT the opening of another New Year I welcome this opportunity of sending a brief message of greeting to readers of THE CHEMICAL AGE.

The year that is past witnessed many achievements, including progress in various spheres of industry and commerce, and especially the relaxation of controls and restrictions. The fertiliser industry is among those industries which have received the boon of greater freedom, and I am proud of the way in which fertiliser manufacturers have seized their new opportunities. As was to be expected from an industry with so fine a record of service, they have shown responsibility and public spirit in their planning under freedom. I have the fullest confidence that these qualities will continue to be demonstrated in all their activities.

The year just beginning opens further fields of opportunity for the manufacturers of fertilisers. The necessity for increased food production from our own farmlands is now linked with that of keeping farm costs of production as low as possible. In both functions the increased use of suitable fertilisers has a vital part to play. So, too, have technical research and the steady advance of applied knowledge in the factory and in the field.

It is to be expected that with the continued demand for maximum food production from British farms the fertiliser industry will receive every incentive to improve on its past records, both in quantity and quality.

I send my sincere greetings and best wishes to all its members and its friends for another successful year of endeavour.

. . . New Year Messages

Mr. E. Hardman

(President, Association of Tar Distillers)

IN reviewing the achievements of 1953 it is necessary to acquaint readers of the wide field covered by the coal tar by-products industry, ranging from bulk products such as creosote, pitch and road tar to the fine chemicals used in medicine, plastics, insecticides, pesticides, dyes, food, preservation, etc. There is hardly an industry in this country, or any other including America, where some of its products are not used.

In its progress during the past 100 years, 1953 being no exception, it has been supported by the Association of Tar Distillers, which is made up of all crude tar producers and distillers of the British Isles. The Coal Tar Research Association, formed five years ago, is now getting into its stride under the able guidance of its director, Dr. McNeil, assisted by a panel from the trade of technical and commercial personnel, and the British Road Tar Association, with its own research in full collaboration with the Department of Scientific and Industrial Research and the Ministry of Transport, renders a very valuable service to all interested in the construction and maintenance of roads in the British Isles.

During 1953 the coal-tar industry has collaborated with the Gas Division of the Ministry of Fuel and Power, the Board of Trade and other Ministries. It has further increased its supply of liquid fuel, the outcome of its valued assistance to the war effort when imported fuels were in short supply. It has met the demand from the Continent and also the home trade for briquetting pitch. It has met in full the demand from the Post Office, railways and private users, for creosote for preserving telegraph poles, railway sleepers, cooling tower timbers, and timbers used for building, agriculture, and general purposes. I think I might claim that coal tar creosote is the finest preservative of timber, this claim being based on its test over the past 80 years. With

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the prospect of some long-overdue attention being given to the country's inadequate road system there is no doubt that road tar will play an increasingly important part in both road construction and maintenance.

As to the future of the by-products industry, it has a definite place in the scheme of things to come, and with the assistance of the Association of Tar Distillers, the Coal Tar Research Association and the British Road Tar Association the industry will give a good account of itself, giving the service its friends expect.

Our achievements will be judged by the service we give, and in sending my greetings for 1954 to your readers, I express the sincere endeavour of the industry I represent to continue to render faithful service to the diverse trades with which it has been so long associated.

* * *

N. Sheldon, A.R.C.S., F.R.I.C.

(Chairman, British Chemical Ware Manufacturers' Association)

AS chairman of the British Chemical Ware Manufacturers' Association, I am glad to have this opportunity of expressing on behalf of members their best wishes to readers of THE CHEMICAL AGE for the New Year.

The manufacturers of laboratory, medical and scientific glassware and porcelain in the United Kingdom have made great strides in increasing their production and in ensuring that adequate supplies are available for the important demand which exists in the United Kingdom and abroad. Their success is shown in the fact that before the war the production of scientific glassware, including both furnace-made and bench-made apparatus in this country, was valued at under £340,000, whereas it had increased just after the war to almost £2,000,000 per annum. In the same period exports jumped from £94,000 to nearly £750,000. Not only in the quantity of production have such strides been made but also in the quality and range of the output of the British industry. As Professor W. E. S. Turner has pointed out, the century has seen a great acceleration of the pace at which glass is being brought into employment for a wide variety of purposes. "The great progress in more recent times has been due to the thorough establishment of the new applied science of glass technology, directed in part to a systematic co-relation between the chemical composition and physical properties of glass, the study of refractories and the development of high temperature processes; in part to the invention of new machinery and processes for converting glass into a vast number of articles of different shapes and sizes; in part to a study of methods of treating glass surfaces; and partly to combining glass with other materials like metals and plastics. This systematic scientific investigation has produced a great range of glasses which can be obtained for many different purposes. It is the object of the manufacturers of glass, silica ware and porcelain to maintain constant touch with users and to make every endeavour to satisfy their requirements. It is realised that science and research must continue to play an important, and increasingly important, part in this country's life and that members of the British Chemical Ware Maufacturers' Association must in their turn play their full part in this movement.

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E. P. Hudson, M.A., F.R.S.E.

(Chairman, Association of Chemical & Allied Employers)

IT is well to recall from time to time the achievements of the British chemical industry in the field of management/labour relationships. The record of initiative and of progress, starting during the first world war, continued through the various difficult phases of the inter-war period, and culminating in the achievements of the industry in the second world war and thereafter, must bring a sense of pride to all in the industry. The pioneering work of our predecessors and the constructive statemanship which the leaders of the industry and of organised labour brought to the consolidation of that work, present a challenge to us in our present circumstances.

How is that challenge being met? Surely we can regard 1953 as a memorable year. We have had the Report of the Heavy Chemicals Productivity Team, one of the

best of a long series, and the impressive follow-up, culminating in the Work Study Conference at Buxton. In the strict field of labour/management relationships I record with special satisfaction the inauguration by the Chemical and Allied Industries National Joint Industrial Council of a comprehensive scheme of training for qualified chemical operators. This scheme, the product of much hard work by representatives of chemical employers encouraged by the enthusiastic support of the signatory trade unions, has evoked a heartening response and many training classes are already at work. In these ways, and others, is shown the effective response of the chemical industry to the challenge of the times.

The year 1953 was a difficult one for chemicals manufacture and trade in many respects. The New Year may bring easement in some directions and greater difficulties in others. Such difficulties have found their reflection in some differences of outlook as to the capacity of the industry to meet a continuation of the pressure for increases in basic wage rate. It is the tradition, and the pride, of all concerned with the operation of industrial relationships in the chemical industry to bring goodwill, good sense and moderation to the council table. That has been so in 1953, and I am confident that the problems of 1954 will be faced in the same spirit.

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Brigadier C. Norton Stafford, C.B.E., T.D.
(Chairman, British Chemical & Dyestuffs Traders' Association)

IT is with pleasure that I accept the editor's invitation to send a New Year's message to the members of the British Chemical & Dyestuffs Traders' Association and our other friends in the chemical and allied trades.

The long period of frustration is now receding, leaving industry and trade with more freedom of action for adapting and readjusting itself to ever-changing world conditions.

Much progress has been made in all sections of the British chemical industry, and the upward trend in the volume of export trade in chemicals during recent years represents an important contribution to the economy of the United Kingdom. The increasing use of chemicals in one form or another for industrial purposes affords a wider scope for the services which the merchant is able to offer to the producer and consumer. With the introduction of new materials and new applications of existing materials, the chemical industry can look to increasing expansion for the future.

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It is by keeping abreast of developments, together with efficient service, and the traditional honest dealing of the British merchant that the trade will sustain its endeavours to meet intensified competition in all markets.

In order successfully to promote our enterprises it is necessary that we should co-operate with each other for our common good, and therefore a representative association is indispensable to any trade of importance. It is through the work of the association, of which I have the honour to be chairman, that the best interests of the British chemical merchants are promoted, and co-operation with other organisations is achieved.

Trading conditions are becoming more stable and there is an optimistic approach to the filling of world markets and the removal of barriers to trade and commerce. Therefore let us enter the New Year with courage and renewed determination to develop our industry to its fullest extent, thus preparing the way for enduring peace and prosperity.

* * *

F. Scholefield, M.Sc., F.R.I.C., F.T.I.
(President, British Association of Chemists)

AN article in a recent issue of the *Board of Trade Journal* began 'The remarkable recovery of German industry in the last three years confronts British enterprise with

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the keenest competition on Switzerland's small but valuable and highly selective market.' This may well serve as the text for what I think should be written with better pens than mine to all British chemists today. For Switzerland's market you may substitute any market, especially for chemical products, and the statement remains substantially true. The British chemical industry in the widest sense is faced with a conflict of skill in science and technology and in salesmanship of the severest kind: the obligation on each one to make his utmost contribution is evident and imperative.

The threat to the maintenance of the position achieved by the British chemical industries during the last eight years is equally a threat to the position achieved by British chemists on the economic plane. Any serious inroad into our chemical production by Germany or by any other competitor must lead to relative insecurity in the positions of, first, industrial chemists, and later, chemists generally.

For more than 30 years, the British Association of Chemists has been concerned chiefly with the economic aspects of the profession, and has been highly successful in its work. In their common interest, all chemists should co-operate in the BAC, and in their individual interest they should put themselves in a position to share the services the Association can render in good times and in bad.

More Sulphate of Ammonia

World Supply Exceeded Demand

WORLD production of pure nitrogen during the 'fertiliser year' ended 30 June, 1953, was more than 11 per cent greater than during the preceding year.

Compiled from published information available and offered as 'fair estimates,' figures given in the annual report of the British Sulphate of Ammonia Association Ltd. show that during the period under review world production of pure nitrogen was 6,138,900 metric tons, compared with 5,515,000 metric tons for the preceding period, and world consumption rose from 5,377,400 metric tons (84.7 per cent as fertiliser), to 6,040,100 metric tons (84.4 per cent as fertiliser).

The estimated European production during the year was 3,266,900 metric tons and consumption 2,806,400 metric tons, 87.7 per cent of which was used as fertiliser. American production is given as 2,145,700 metric tons and consumption as 2,109,000 metric tons.

Industrial usage throughout the world was 14½ per cent higher than during the previous period, while consumption for agricultural purposes went up by nearly 12 per cent. The report points out, however, that although total consumption showed a higher rate of increase than production, total supply exceeded demand—as in the previous year—so that there was a small addition to world stock.

Exports of sulphate of ammonia from the United Kingdom during the year exceeded 455,000 tons, this being an increase of 31 per cent over the high total reached the previous year.

In a reference to sulphate of ammonia and agricultural development in the UK, the report states:—'The development work on grassland done by our agent, Imperial Chemical Industries Limited, was continued and extended with gratifying results. A full programme of demonstrations and conferences was carried through successfully and many thousands of farmers benefited from the advice and factual presentation given. This work is receiving official support from the National Agricultural Advisory Service of the Ministry of Agriculture.'

Reference is made to manufacturing research as follows:—'Work on the development of suitable analytical methods for the determination of impurities in saturator liquors has continued and the laboratory staffs of several of our members have collaborated in this work.

'The use of resin cements and lacquer for protective coatings continue and very promising results are being obtained.'

Developments in the Use of Primary Standards & Indicators

by A. J. NUTTEN, B.Sc., Ph.D., A.R.I.C.
(The University, Birmingham)

DURING the past two years a fair amount of attention has been concentrated on the development of new primary standards and new indicators. In addition several well-established primary standards have been re-examined in order to clear up doubtful points concerning them, and the usefulness of some conventional indicators has been extended by finding new applications for them in titrations in which they have hitherto not been used.

The following survey covers some of these recent investigations.

PRIMARY STANDARDS

Standards of Acidimetry & Alkalimetry

Smith[1] recommended potassium hydrogen *bis*(3.5 - dinitrobenzoate) $KHC_{14}H_6O_{12}N_4$ as an alkalimetric standard. It is readily prepared by mixing 212 gm. of 3,5-dinitrobenzoic acid dissolved in 60 ml. of acetone with 3.5 gm. of potassium carbonate dissolved in 20 ml. of hot water and recrystallising from 3 : 1 acetone-water.

The product is dried in a vacuum desiccator. It is non-hygroscopic and has been kept for six years without noticeable change. Its equivalent weight is 462.3.

Although the standard is not very soluble in water, the latter decomposes it to the free acid and an aqueous solution containing the potassium salt and a little free acid.

The dissociation constant of the acid is 1.57×10^{-3} at 25°, and the most suitable indicator for the titration with alkali is thymol blue. Smith claims that this new standard has many advantages over the conventional organic acid standards.

Sulphamic acid has been used as a primary standard in alkalimetry for some years, but its solutions slowly hydrolyse at room temperatures, although it has been suggested that the titre should remain constant because the bisulphate ion is formed:

$$NH_2SO_2OH + H_2O \rightarrow NH_4HSO_4$$

and there should be no change in acidity. Accordingly, Wagner, Wuellner and Feiler[2] have examined the constancy of titre of sulphamic acid solutions.

After 213 days there was no significant change in the titre when the solutions were stored in borosilicate glass bottles protected from carbon dioxide by tubes containing Ascarite. After six days there was a slight test given for the sulphate ion which became more pronounced as the storage time increased. When pure sodium carbonate was checked against sulphamic acid as primary standard the claims already made were supported.

Although potassium hydrogen phthalate is widely accepted as a primary alkalimetric standard some disagreement exists as to its thermal stability. Hendrixson[3] states that it is stable up to 150°, but Kolthoff and Stenger[4] advise against heating above 125° because of possible loss of phthalic anhydride. Hillebrand and Lundell[5] recommend 120° whereas Willard and Furman[6] among others specify 110°. The National Bureau of Standards recommends drying at 120° for one to two hours. Because of these conflicting statements Caley and Brundin[7] have recently studied the thermal stability over the range 110° to 200°.

Thermal Decomposition

It was found that thermal decomposition first becomes noticeable around 145° and becomes fast enough for convenient measurement at 150°. The first products of decomposition appear to be the normal salt and phthalic acid but the latter probably decomposes immediately into phthalic anhydride and water. The compound could be heated at 135° without risk of decomposition and this temperature is recommended as the safe upper limit.

Samples heated at 110° until no further loss occurred lost a further 0.01 per cent when heated in the range 130° to 140°. Although this loss may have been due to water held tenaciously in the crystals, since these had not been ground, it was considered that a more likely explanation was that

some volatile substance or substances such as water and phthalic anhydride produced by decomposition of phthalic acid—a common impurity in potassium hydrogen phthalate—caused the loss. The authors did not, however, check this by noting if the finely powdered salt behaved likewise. If this loss be due to volatile products it would appear necessary to heat the salt always at 130° to 135° to ensure their elimination.

Sulphuric Acid

Kunzler[8] has described various ways of preparing sulphuric acid as an alkalimetric and acidimetric standard. For the highest accuracy he recommends preparation by a maximum freezing point method, but for ease of preparation constant boiling sulphuric acid is to be preferred. The latter has a number of advantages. It can be prepared with an accuracy of at least 0.01 per cent, which is superior to that obtained with constant boiling hydrochloric acid. The composition of the latter changes one part in 10,000 for a pressure change of 1 mm. For a similar pressure change, sulphuric acid changes only one part in 400,000. Other advantages are that sulphuric acid is readily available in a fairly pure form and can be further purified by a simple distillation, and although hygroscopic it is non-volatile. Moreover, a liquid standard is to be preferred because solid standards can have solution inclusions. Kunzler also claims that constant boiling hydrochloric acid if not standardised against a good primary standard may give errors as much as 0.1 per cent.

In spite of these claims the preparation of sulphuric acid for use as a primary standard is by no means attractive and it is extremely doubtful that it will supplant conventional solid primary standards.

Shead[9] has advanced calcium hydrogen maleate hexahydrate as a several purpose primary standard. It has a very high equivalent weight (207.143), is readily obtained pure and when properly bottled shows no sign of gain or loss in weight. It cannot be dried at 100° which is its one disadvantage. After crystallising, the sample may be air-dried if the humidity is low, but in humid climates dry air should be cautiously drawn over the crystals.

The compound is recommended for the standardisation of alkali titrants, for complexone solutions used in titrations of calcium, and for the calibration of *p*H meters. If it is calcined to calcium oxide in a platinum dish it can be used to standardise acid titrants by solution in excess and back-titration with a base having a known ratio to the acid. Finally, it may have some use for standardising Karl Fischer reagent because of its constant content of water of crystallisation, if a suitable anhydrous solvent could be found.

The properties of acetylsalicylic acid as a primary alkalimetric standard have been investigated by Burriel, Pino and Vinuesa[10]. The compound cannot be dried by heating but it is practically non-hygroscopic and crystallises without water. When the phenolphthalein end-point is reached with 0.1N sodium hydroxide, the colour fades owing to the formation of salicylic acid. The fading of the end-point does not interfere with the determination. Hydrolysis does not occur before neutralisation, as can be shown by the ferric chloride test for free salicylic acid which is sensitive to 0.05 mg. The hydrolysis can be completed by heating with excess alkali and back titrating, which results in a further consumption of alkali. This procedure is used to check the purity of acetylsalicylic acid for use as a primary standard.

It is necessary to dissolve the standard initially in 50 per cent ethanol.

The results with this new primary standard agreed to about one part in 1,000 when compared with other primary standards.

Many Desirable Properties

2,4,6-Trinitrobenzoic acid has been examined as an alkalimetric primary standard by Smith and Wilkins[11]. It appears to have many desirable properties. The pK_a value is 2.38, it has a high equivalent weight (257.12), is readily purified, is anhydrous and stable at 130° or in air for weighing and storage. An unusual property is that it serves as its own indicator (colourless to red at about *p*H 8). When titrated with 0.1N alkali, the acid gives an abrupt *p*H change over the range 4.5-8.5 with the addition of a very small amount of alkali. This change compares favourably with that given by sulphamic acid and the *p*H change is more pronounced. The end-point is best marked with bromothymol blue which changes nearest to the theoretical equivalence-point.

The standard is prepared from the technical grade acid (330 gm.) by slowly adding

15 per cent sodium hydroxide solution to the acid suspended in water (2,000 ml.) at 35°, using a mechanical stirrer. Upon complete conversion to the soluble sodium salt, the solution turns pink and this point should not be exceeded. The pink colour is then just cleared by the addition of a few drops of acetic acid. The insoluble impurities are then filtered off and washed a few times with water. This insoluble material is mainly 2,4,6-trinitrotoluene.

The filtrate is then acidified with 50 per cent sulphuric acid, chilled, filtered and washed with water until free from sulphuric acid. The yield is 60 per cent. The purification is best repeated, particularly if the starting material is crude. The crystals are air-dried, then placed in a desiccator over Anhydrone and finally are heated in an oven at 85°.

The titration values compare well with those given by potassium hydrogen phthalate and sulphamic acid.

A Stable Standard

Tris-(hydroxymethyl) aminomethane has been recommended as a primary acidimetric standard by Fossum, Markunas and Riddick[12]. Its equivalent weight is 121.36. They compared it against hydrochloric acid standardised against two commonly accepted standards, sodium carbonate[9], and sodium hydroxide solution standardised potentiometrically against potassium hydrogen phthalate. Excellent agreement was obtained.

There was no detectable change in composition of the new standard after two years' storage in a stoppered bottle. No change in weight could be observed after storage over phosphorus pentoxide in a desiccator for three days. Tests on the thermal stability of the compound indicated that it could be heated for two to four hours at 100° to 103° without decomposition. Above this temperature decomposition begins. The hygroscopicity is approximately that of potassium hydrogen phthalate, and it does not absorb carbon dioxide from the air either as the solid or in solution. The compound is a weak base and has a dissociation constant of 1.202×10^{-6} at 25°.

Suitable indicators to use in titrations with the standard are *p*-sulpho-*o*-methoxybenzene-azo-dimethyl - 1 - naphthylamine, ethyl orange and a screened indicator consisting of bromocresol green and sodium alizarin sulphonate, all of which give a sharp colour change at *p*H 4.7. The first indicator is to be preferred, however, for it gives the sharpest change and gives a good warning of the approaching end-point.

Standards for Silver Nitrate

The usual methods for purification of reagent-grade sodium chloride for use in argentimetry involve either several recrystallisations from water or precipitation by hydrochloric acid gas. The former process is tedious and the yield is poor; the latter requires a generator and only a small amount of potassium salts is removed from the original material. Meites[13] has described a new simple method which gives a 70-80 per cent yield with an effective purity of 100 ± 0.01 per cent. The product is free from sulphate, calcium, magnesium, barium, bromide and iodide, as shown by the tests recommended by Kolthoff and Stenger. Spectroscopic examination showed that lithium and zinc were the only detectable impurities.

A two-stage precipitation with hydrochloric acid is used. In the first only a small amount of hydrochloric acid is added to the nearly saturated solution. This precipitates any insoluble material originally present and most of the potassium chloride and bromide. Pure sodium chloride is then precipitated by adding a large amount of hydrochloric acid. The product is filtered, washed, air-dried by suction and finally dried at 180°.

Standards for Redox Titrations

The requirements for primary redox standards have been advanced recently by Stenger[14]. He refers to the fact that Fowler and Bright[15] showed that permanganate titres are 0.2-0.4 per cent high when the conventional heating procedure is used, owing to decomposition of oxalic acid. When most of the permanganate is added in the cold and the solution is then heated, much better results are obtained. Thus, although sodium oxalate is a pure reproducible substance, it was an unreliable standard until the stoichiometry of its use had been established. Because of the complicated reactions involved, Stenger does not consider sodium oxalate to be the best standard for permanganate. When used under specified conditions, arsenious oxide is a better standard than sodium oxalate.

After considering other well-known standards, Stenger summarises the following requirements:

(1) A primary standard must be stable enough to permit accurate weighing and ordinary analytical manipulations.

(2) The purity of a primary standard must be assumed: through well-defined qualitative tests of known sensitivity, or through preparation by a method that has been demonstrated to yield a pure product consistently, and storage under conditions in which the product is entirely stable.

(3) The suitability of a primary standard for a particular purpose must be demonstrated by stoichiometric studies. These studies must be carried out as accurately as possible, but need be made only once for each case.

Standards for Iodine

Barium thiosulphate was first suggested as a primary standard in iodimetry by Plimpton and Chorley[16] in 1895. They pointed out that it was easily prepared in a pure state, it was stable and it had a high equivalent weight (267.50). The one disadvantage was its low solubility (2.66 gm./litre at 25°). Several workers have examined the compound as a standard since that time but a number of uncertainties in its use still remain to be answered. For this reason MacNevin and Kriege[17] have subjected barium thiosulphate monohydrate to a searching examination and they report very favourably on its use as a primary standard. The only disadvantages are its low thermal stability and low solubility, but these cause only slight inconvenience.

Warm water solutions of barium chloride and sodium thiosulphate were mixed, to prepare the standard. The crystalline precipitate filtered readily and was washed with ethanol, followed by ether. The latter was removed by suction, leaving the pure monohydrate. The compound is stable at room temperature but its thermal decomposition was studied, using a thermobalance. It was stable up to 50° but between 50° and 100° there was a large loss. Further losses occurred between 100° and 200°. The safest permissible temperature which can be used for heating the compound is 40°.

The anhydrous compound was also examined but it dissolves too slowly to be of service. The monohydrate, however, dissolves as the titration with iodine proceeds and there is no delay owing to slow solution. Indeed, the disappearance of the last trace of solid gives a warning of the approach of the end-point.

It was established that the average purity of the samples prepared was 99.85 per cent.

Standards for the Karl Fischer Reagent

Although several hydrates have been recommended as standards for the Karl Fischer reagent, no details as regards stability or establishing the accurate water content have been given. Accordingly, Neuss, O'Brien and Frediani[18] examined a wide range of hydrates to find a standard which would fulfil the following conditions:

(1) It should be readily available in a pure form and contain the theoretical water content.

(2) It should react rapidly and quantitatively with the titrant and give a sharp end-point in methanol solution.

(3) It should be stable in ordinary containers for long periods under extreme conditions of humidity.

(4) Its water content should be subject to independent check by some convenient and accurate method, such as determination of loss by heating.

The following hydrates were examined: Aluminium potassium sulphate, ammonium oxalate, citric acid, ferric ammonium sulphate, ferrous ammonium sulphate, lactose, oxalic acid, potassium citrate, potassium tartrate, sodium acetate, sodium bitartrate, sodium tartrate, sodium citrate, sulphosalicylic acid.

Of all the substances tested, sodium tartrate dihydrate proved to be outstanding. Its only defect is insolubility in methanol, but this in no way impairs the sharpness of the end-point. This compound was finally selected and has given very satisfactory results over a period of three years.

INDICATORS

Indicators for Titration in Non-aqueous Media

Riddick[19] has recently discussed acid-base indicators for use in titrations in non-aqueous solvents. He recommends xylene cyanol, methyl red, thymol blue, methyl violet, phenolphthalein, 1-naphtholbenzein, bromocresol yellow, crystal violet, benzylauramine, methyl orange and cresol red. Fritz[20] uses azo-violet or thymol blue in

titrations of ammonium salts of mineral acids, aliphatic amines or bases. Ethylenediamine or dimethylformamide is used as solvent and sodium methoxide in benzene-methyl alcohol as the titrant.

For titrations in glacial acetic acid based on the formation of insoluble salts, Tomicek and Zukriegelova[21] recommend potassium nitroprusside or dithizone for the titration of lithium bromide or chloride with mercuric acetate, neutral red, thionine or safranine using lead acetate as titrant, and neutral red or thionine using thallium acetate as titrant.

Seaman and Allen[29] examined the properties of crystal violet as an indicator for acid-base titrations in glacial acetic acid using perchloric acid as titrant. They titrated sodium carbonate, glycine, potassium hydrogen phthalate and methyl nicotinate, and found that the colour change at the end-point varied according to the system titrated. They recommended therefore that the end-point for any particular system be first assessed potentiometrically and the particular colour change for crystal violet at that stage be taken for future visual titrations. Colour reference solutions are recommended in some cases.

Redox Indicators

Adams and Hammaker[23] have investigated the usefulness of Alphazurine G as a redox indicator. The transition potentials in various concentrations of sulphuric, hydrochloric and perchloric acid were determined for the titration of ferrous iron and ferrocyanide with cerate. The indicator is recommended for the cerate-ferrous but not for the cerate-ferrocyanide titration. The same workers have also proposed two new redox indicators, N.N′-tetramethylbenzidine and N.N′-tetramethylbenzidine-3-sulphonic acid. N.N′-tetraethylbenzidine was also examined but was found to be unsatisfactory as a redox indicator. The transition potentials of the two new indicators were determined as in the case of Alphazurine G.

The colour change of both indicators was from colourless to deep yellow and a warning was given of the approach of the end-point by the development of a faint pink colour, 0.5-1.0 ml. before the deep yellow colour appeared. In dilute solution the base was better than its sulphonate, but the latter was preferable in perchloric acid solution. The base is stable even at the boiling point of the solution which suggested it may be of service for titrations at elevated temperatures.

For the reductometric titrations with ascorbic acid, Erdey and Bodor[26] recommend 4-amino-4′-methoxy-diphenylamine. The oxidation of the indicator is a complex process which occurs in two stages. The oxidation potential is dependent on the *p*H of the solution and it is concluded that the indicator functions best at *p*H 6. At *p*H 6.7 titration is still possible but the end-point is difficult to detect. In alkaline solution no end-point at all can be observed.

Titrations with Ascorbic Acid

In titrations of ferric iron with ascorbic acid, the new indicator is better than potassium thiocyanate, although the results obtained are slightly lower. The indicator is best added near the end-point to prevent irreversible oxidation and the solution must then be heated to 40-50°.

The new indicator may also be used in the titration of dichromate, vanadate, silver, gold and platinum with ascorbic acid, titanous chloride, stannous chloride and chromous sulphate, and the oxidimetric titrations with ceric sulphate and chloramine-T. Iodimetric titrations are possible of th *p*H be adjusted to 2-6.

In the determination of ferrous iron with dichromate it is necessary to add potassium fluoride. The results obtained are in good agreement with those obtained potentiometrically but diphenylamine gives slightly higher titres.

The application of *ortho-*, *meta-* and *para*-tolyl-anthranilic acids as redox indicators has been studied by Lederer and Ward[25]. They reached the following conclusions:

(1) A methyl group in the *ortho* position increases the redox potential.

(2) A methyl group in the *meta* position decreases the redox potential. The oxidation products are unstable.

(3) A methyl group in the *para* position prevents oxidation to diphenylbenzidine blue.

The *ortho*-derivative is the only one suitable for use as a redox indicator. It gives a stable oxidation product and the colour change (to purple) is very similar to that of N-phenyl-anthranilic acid. Although the *meta*-derivative gives a blue colour, it is very unstable and fades almost immediately.

Milner[26] recommends naphthidine as indicator for the determination of zinc in a wide range of non-ferrous alloys and in rubber ash by titration with ferrocyanide. He found this indicator preferable to those used previously in this titration.

Adsorption Indicators

Manelli and Rossi[27] have recommended Red Acid 6B as an adsorption indicator in the argentometric determination of iodide ion. They consider that this indicator is very suitable for very small quantities of iodide. The same authors also recommend Blue Sulphone Acid B28 for the titration of iodides in an alkaline medium and further suggest that this indicator might be suitable as an acid-base indicator between *p*H 11 and 12.

At the equivalence point for the titration of bromide with silver ions, the potential of the system is 1.43 + 0.03 log [Br_2]. Rancke-Madsen[28] deduced that the transition interval for a suitable indicator should be 1.30-1.35 volt, and he examined nitro-ferroin which can be used in the range 1.30 volt, with a small amount of free bromine present. At the equivalence point there was a change from red to yellow on the precipitate. Although this transition was very sharp it was difficult to observe in artificial light. Chloride and iodide must be absent, which limits the application of the method. It is, therefore, mainly of theoretical interest.

Miscellaneous Indicators

The determination of chloride by titration with a mercuric salt is usually effected with diphenylcarbazide or diphenylcarbazone as the indicator. Both indicators have the disadvantage that the colour change is gradual. Parsons and Yoe[29] have shown that diphenylcarbazide disodium disulphonate gives a better end-point. Ferric ions are added just before the titration to oxidise the indicator to the diphenylcarbazone sulphonic acid together with nickel nitrate to screen the colour change which is otherwise from yellow-orange to violet. The *p*H of the test solution is adjusted to *p*H 2 with 0.4N nitric acid before adding the mixed ferric-nickel nitrate solution. A warning of the end-point is obtained by the appearance of a neutral grey shade from the original green and the titration is then continued to a purple hue. One drop of titrant turns the solution from green to grey and a fraction of a drop to the purple shade.

In the determination of water hardness by titration with complexone, Eriochrome Black is used as indicator in the titration of magnesium. This indicator has the disadvantage of decomposing fairly rapidly in ethanolic solution, the conventional solvent, and it is necessary to prepare fresh solutions quite frequently. Diskant[31] examined a wide range of solvents and found that diethanolamine or triethanolamine gave the most stable solution. After seven months there was no sign of deterioration even though no precautions were taken to protect the solutions from light.

The conditions for use of dithizone as indicator in zinc-ferrocyanide titrations have been established by Mehlig and Guill[32]. They find it best to back-titrate with a standard zinc solution because only an excess of ferrocyanide would discharge the indicator colour. The complex is stable between *p*H 3.5-5.0; it turns from green to pink in the presence of zinc ions.

The method was applied to the determination of zinc in sphalerite ores and gave excellent results when compared against the gravimetric method.

Ferroin has been used as indicator in the titration of mercuric and bromide ions[33]. Mercuric ion is titrated directly with bromide to the appearance of a faint pink turbidity. Bromide is best titrated by adding a known excess of mercuric nitrate and back-titrating with standard potassium bromide solution. Chloride and iodide interfere.

The principle of the titration depends on the following equations:

$$Hg^{++} + 2Br^- \rightarrow HgBr_2$$

$$HgBr_2 + Br^- \rightarrow HgBr_3^-$$

$$2HgBr_3^- + (C_{12}H_8N_2)_3Fe^{++} \rightarrow (C_{12}H_8N_2)_3Fe(HgBr_3)_2$$

The complex formed is an insoluble red compound which can also be used for the gravimetric determination of mercury.

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The Hunt is On

FIRST explorations in the Gas Council's £1,000,000 search for natural gas, which will extend over the next five years, are now taking place in the East Riding of Yorkshire. A seismic survey, which investigates the nature of the underground rock structure to determine whether supplies of natural gas are likely to be found in the area, is proceeding on a line from Aldbrough to Market Weighton. The photograph below was taken in a field near Whitedale station about six miles inland from Aldbrough. The rock structure is measured by firing a charge of dynamite in a shot hole drilled to a depth of about 60 to 70 ft. The seismic waves are reflected from the various rock structures and picked up by sensitive instruments, known as geophones, placed in the ground at fixed intervals along the line of the survey. By comparing the results obtained on the different instruments the nature of the structure can be calculated.

Running out the cable which connects the geophones to the recorder. The technician in the foreground has geophones ready to place in the ground and connected to the cable

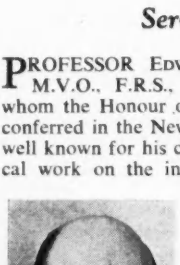
First Instrument Congress

THE First International Instrument Congress and Exposition will be held in Philadelphia, USA, from 13 to 25 September, 1954. Laboratory apparatus and equipment, as well as measuring and control instruments of every kind, will be available for inspection and discussion. The Instrument Society of America, in its capacity of organiser of the Congress and Exposition, has invited a large number of technical and scientific societies, both in the United States and elsewhere, to take an active part in the organisation of this notable event. The DECHEMA (Deutsche Gesellschaft für chemisches Apparatewesen), as one of the societies invited to participate in the Congress, is proposing to organise two scientific journeys to the United States in connection therewith. It is proposed that one journey shall have a duration of six days, while the other will extend over a period of four weeks.

New Year Honours

Services to Science & Industry Rewarded

PROFESSOR EDWARD CHARLES DODDS, M.V.O., F.R.S., M.D., F.R.C.P., upon whom the Honour of Knighthood has been conferred in the New Year Honours, is very well known for his comprehensive biochemical work on the internal secretions of the body. Born in 1899, he has been Courtauld Professor of Biochemistry in the University of London since 1924, and director of the Courtauld Institute since 1925. Among the many honours which have been conferred upon him in the course of a distinguished career are the Garton Prize and Medal, British Empire Cancer Campaign, 1948; Berzelius Medal, Swedish Medical Society; Pasteur Medal of VIIIth Congrès de Chimie Biologique de la Société de Chimie Biologique Francaise; the Gold Medal of the Society of Chemical Industry, 1951; and the Harben Medal of the Royal Institute of Public Health, 1952.



Knighthoods have also been conferred upon GEORGE FOSTER EARLE, C.B.E., who has been chairman of the Associated Portland Cement Manufacturers Ltd. since 1948; CECIL GEORGE GRAHAM HAYMAN, director and chairman of the Management Committee, Distillers Co. Ltd., and immediate past-chairman of the Association of British Chemical Manufacturers; OLIVER LYLE, O.B.E., for services in promoting fuel efficiency; and EDWARD TALBOT PARIS, C.B., chief scientific adviser to the Home Office since 1948, and formerly director of research at the Ministry of Supply.

Other recipients of honours include the following:—

Knight Commander of the British Empire

The Right Honourable JOHN CAMPBELL, BARON TRENT, J.P., D.L., who is retiring from the chairmanship of the Boots Pure Drug Co. at the end of January owing to ill health (see CHEMICAL AGE, **70**, 38).

Commanders of the Order of the British Empire

WILLIAM KENDALL DAVEY, chairman of the London Metal Exchange; ST. JOHN DE HOLT ELSTUB, director of the Metals Division, I.C.I.; JAMES BRIERLEY FIRTH, director of the North Western Forensic Science Laboratory; WILLIAM KENNETH HUTCHISON, chairman of the South Eastern Gas Board; ERNEST THOMAS OSBORNE, O.B.E., director of chemical inspection, Ministry of Supply; BERNARD ALFRED SOUTHGATE, director of the Water Pollution Research Laboratory; and AUBREY JOHN CUMING WALTERS, director and secretary of the British Rayon and Synthetic Fibres Federation.

Officers of the Order of the British Empire

HENRY NORMAN BINNS, general manager of the Great Grimsby Coal, Salt and Tanning Co. Ltd.; DAVID DOUGLAS BURNS, deputy chairman of the Scottish Gas Board; ROGER KENDAL CUSHING, principal scientific officer at the Ministry of Supply; ALFRED VICTOR DALZELL, secretary-director of the Federated Quarry Owners of Great Britain; ROBERT BALDWIN HOVEY, M.C., chairman of Zan Ltd., Sandbach; HOWARD GEORGE HENRY KEARNS, senior principal scientific officer, Long Ashton Research Station; THOMAS WARBURTON MCCULLOUGH, lately Superintending Inspector of Factories in Scotland, now deputy Chief Inspector of Factories, Ministry of Labour and National Service; ARNOLD MARSH, director of the National Smoke Abatement Society; PHILIP EDWARD MONTAGNON, senior principal scientific officer to the Ministry of Fuel and Power; BULLER ALFRED RENTON, principal at the Ministry of Materials; FREDERICK GEORGE SKINNER, deputy keeper, Science Museum; and ARTHUR GEORGE TARRANT, senior principal officer, Road Research Laboratory.

Members of the Order of the British Empire

JOHN STEPHEN BALL, head of design and development at Henry Simon Ltd., Stockport; HERBERT FRANCIS WILLIAM GOLDING, chief inspector for the Northern Aluminium Co. Ltd., Banbury; WILLIAM CHARLES JOHNSON, chief chemist, Baird & Tatlock (London) Ltd.; and MISS MILDRED BEATRICE WATERS, higher executive officer, DSIR.

Recent Developments in Dairy Chemistry

by F. H. GRIMBLEBY, B.Sc., Ph.D.
(Department of Agricultural Chemistry, The University, Reading)

THE demand for liquid milk and for milk products of all kinds has increased considerably during the last decade, due to the progressive expansion of the world's population and the higher human nutritional levels generally attained or demanded. Still further demands have been made by those industries which consume a considerable amount of casein for the production of plastics, textiles, fibres, glues, etc.

These demands have resulted in an increase in the number of dairy herds maintained and the widespread adoption of a policy of selective breeding which has resulted in a progressive increase in the efficiency of the cow as a converter of fodder to milk. Further, these demands have been responsible for a considerable increase in the amount of fundamental research carried out on milk and its products with a view, finally, to improving the various methods of processing and the elimination of waste resulting from deterioration, particularly in dried stored products.

The volume of literature published during the last few years which has been devoted to the chemistry of milk and its products has been very considerable and it is not possible in this review to give detailed consideration to all of it. It is proposed, therefore, to deal with a number of selected subjects upon which work has been done extensively and where the results of the work present a clear-cut picture of the subjects studied.

THE COMPOSITION OF MILK

Information regarding the compositional quality of milk has been supplied by a number of workers in different parts of the world and it is of interest to note that similar trends have been noted in all countries for which records are available. Provan[1], Davis[2] and Bailey[3] have each examined records collected in England and Wales over periods up to 50 years and while Davis found that the fat and solids-not-fat contents of milk had increased from 1900 to 1930, all three investigators found that there had been a progressive fall in solids-not-fat since about 1930.

Grigg[4], at Christchurch, New Zealand, found that while the fat content of milk had hardly changed at all during the period 1926-45, solids-not-fat had declined. Bakalor[5] reported a similar decline in the solids-not-fat content of South African milks and Eyrard, Hurel and Boisseau[6] found a general decline in solids-not-fat contents of milks, collected from all parts of France, from 90 gm. per litre in 1943 to 88 gm. per litre in 1945-46.

Effect of Diet

Bakalor has suggested that the fall in compositional quality in South Africa is due partly to the replacement of indigenous cattle by imported cattle giving higher yields but a lower concentration of milk solids, while Grigg considers the general decline in New Zealand to be due to a breeding policy based on yield and butterfat content of milk. Rowland[7] has shown that the feeding of low energy and/or low protein diets to dairy cows is frequently responsible for poor compositional quality and that the decline in the solds-not-fat of milk during the war years was due chiefly to this cause.

The effect of the nature of the cow's diet on the composition of milk has been investigated by McClymont and Paxton[8] and McClymont[9] who noted a considerable fall in the fat content of milk when cows were fed on a diet containing an abnormally low percentage of crude fibre. Increased quantities of roughage resulted in the fat content of the milk returning to normal.

Studies devoted to the synthesis of milk fat in the lactating mammary gland by Folley and his associates have since provided an explanation of this important finding. The milk fat of cows differs from the fat in the circulating blood in that it contains fatty acids of low molecular weight and it must be presumed that in addition to the blood fat which is taken up without alteration by the milk gland, a considerable amount of milk fat is synthesised by the milk-secreting cells. The origins of these fatty acids of low molcular weight has

received much attention and three possible modes of synthesis have been suggested, viz.,

1. The degradation of long chain fatty acids.
2. Synthesis from carbohydrate.
3. Synthesis from short chain acids produced in the rumen of ruminant animals.

There has never been much evidence in favour of the first suggestion and it has been finally discounted by the findings of Popjak, French and Folley which are detailed below. The suggestion that fat was synthesised from carbohydrate received little support on the grounds that after the utilisation of absorbed blood glucose for lactose synthesis there is little, if any, available for the synthesis of fat. Folley and French[10] have further shown that while glucose is utilised for fatty-acid synthesis by non-ruminants, the mammary tissue from ruminants is almost inert towards glucose.

Utilisation of Rumen Acids

The third suggestion that ruminants utilise acetate for fatty acid synthesis has received a considerable amount of experimental support. It is significant that appreciable amounts of short chain acids are not found in the milk fat of mammals other than ruminants, and the conclusion must be drawn that rumination is responsible for the supply of the precursors of the short chain acids found in ruminant milk fat. Elsden[11] has shown that fermentation of food in the rumen results in the production of considerable amounts of acetic acid and smaller quantities of propionic and butyric acids. Subsequent absorption of these acids from the rumen, reticulum, and omasum loads the circulating blood with these acids and provides the mammary gland with a substrate which has been shown by Folley and French to be eminently suitable for the synthesis of fat.

Popjak, French and Folley[12] have also shown that when acetate, with the carboxyl carbon labelled with ^{14}C, was injected intravenously into a lactating goat, the ^{14}C was recovered almost entirely in the volatile fatty acids of the milk fat, indicating that these volatile acids could not have originated from the degradation of long chain fatty acids but must have been synthesised from acetate or a derivative of it.

The findings of McClymont and Paxton are explained by the fact that the feeding of roughage to ruminant animals delays the passage of food from the rumen thereby permitting extensive fermentation of carbohydrate to acetate to occur; if the diet contains insufficient roughage, the food passes from the rumen before acetate is produced in appreciable amounts, the mammary gland is deprived of the substrate necessary for the synthesis of fat and the fat content of the milk is consequently reduced. The influence of rumen fermentation on the fat content of milk was further indicated by the experiments of Tangl and Szarka[13] who obtained an increase in the fat content of milk by feeding fresh yeast but no increase on feeding dried yeast to lactating cows.

Seasonal variations in the composition of milk have been studied by Kastli and Binz[14] in Switzerland who found that both fat and solids-not-fat increased from minimum values in March to maximum values in October with a difference of 0.05 per cent between the maximum and minimum values for fat and 0.15 per cent for solids-not-fat. Eyrard *et al* found that in France the minimum for solids-not-fat was reached in August and the maximum in early winter. Rensburg[15] found that the solids-not-fat of South African milks was highest in October-December and lowest in June-August. These findings are in general agreement with those of earlier workers.

Freezing Point Depression

Genuine milks having very low contents of milk solids and displaying abnormally low freezing point depressions have been described by several workers, notably Koenig[16] who found that the cows secreting the abnormal milk were being maintained on a semi-starvation diet. When they were fed indoors on fresh-cut grass they gave milk of normal composition and freezing point.

Hillman, Provan and Steane[17] and Aschaffenburg and Rowland[18] investigated genuine but abnormal milk samples produced by cows at the evening milking only. They found that the cows were kept indoors without water for 16 hours during the night and imbibed copious quantities of water when given access to it the following morning. This, coupled with the feeding of mangolds, resulted in the production in the evening of milk which had all the characteristics of a watered sample. The abnormality disappeared when the cows were allowed access to water all day.

Bailey[3] has made a close examination of the herd records collected at the National Institute for Research in Dairying over the 12-years period from 1935-46. The data indicate that highest quality milk was produced by first calvers and the highest yield by sixth calvers. No correlation was found between lactation yield and solids-not-fat percentage and the correlation between lactation butterfat and solids-not-fat percentage was so low as to suggest that a breeding policy to improve milk quality should not be based on butterfat records.

THE CONSTITUENTS OF MILK

1. *Milk Fat*

The presence of octadecadienoic acids in milk fat has been established by Hilditch and Jasperson[19] and determined by Schaffer and Holm[20] who found 2.62-2.71 per cent linoleic acid in summer milk fat and 2.11-2.31 per cent in spring and winter milk fats; these workers also found 0.72-1.29 per cent of octadecatrienoic acids in milk fat. The vaccenic acid content of milk fat is given as 1.28 and 4.93 per cent by Brouwer and Jonkin-Scheffener[21], the higher value being obtained by pasture feeding in spring and the lower value on a winter ration. Geyer, Nath, Barki, Elvehjem and Hart[22] have recorded values of 0.5-0.7 per cent.

Due to its importance in influencing butter texture and its effect on the rate of deterioration of fats through the development of taints by oxidation, the iodine value of milk fat has received a considerable amount of attention. The influence of the character of the ingested fat on the iodine value of milk fat has been investigated by Hilditch and Jasperson[23] and also by Anantakrishnan, Bhalerao and Paul[24] who have shown that there is a general tendency for the IV of milk fat to change in the direction of that of the ingested fat. Increases in the IV due to a higher content of oleic acid were generally accompanied by a decrease in the Reichert, Polenske and Kirschner values.

Sjollema[25] has made the suggestion that the iodine value of milk fat is raised by increases in the crude protein content of the diet, but this is discounted by the experiments of Jarl[26] who found significant decreases in the IV of milk fat when cows were fed excessive quantities of digestible protein in the form of dried skim milk.

The chemical reactions involved in the autoxidation of unsaturated fatty acids in fats have been studied and reviewed by Skellon and Gordon[27] who have concluded that the primary oxidation product of monoethenoid acids is the monohydro-peroxide formed by the addition of oxygen to the methylene group contiguous to the double bond.

According to Hilditch[28] monoethenoid acids are resistant to oxidation at temperatures below 50° in the absence of more highly unsaturated compounds and he considers that the initiation of oxidation is a function solely of the long chain unsaturated acids which contain the pentadiene system $-CH=CH-CH_2-CH=CH-$ with a central reactive methylene group. Active peroxide radicals produced by the addition of oxygen to this reactive group then propagate a chain reaction involving monoethenoid acids. The hydroperoxides formed by this chain reaction are not very stable and decompose with the formation of epoxy, aldehydic, dihydroxy or ketohydroxy derivatives. Prolonged oxidation results in dimerisation and polymerisation, but the exact point at which polymers form and their exact rôle remains to be investigated.

Stale Flavour Components

Whitney, Paulson and Tracy[29] have separated the stale flavour component from stale butter oil by steam distillation followed by extraction of the distillate with petroleum ether. The petrol-soluble fraction, when dissolved in fresh butter oil and blended with fresh products to the composition of the original milk, yielded a detectable stale flavour in concentrations as low as 6.2. ppm. Keeney and Doan[30] separated the volatile constituents of oxidised fat by vacuum distillation and found that the predominant odour compounds were ketones, none of which were methyl ketones while a large proportion of them were unsaturated.

Carbonyl compounds having the empirical formulæ $C_3H_{4-6}O_2$, $C_7H_{12}O$, $C_9H_{16}O$ and $C_{12}H_{20-22}O$ were separated, the first compound being identified as 2-hydroxypropanal, the C_7 and C_9 compounds as unsaturated ketones and C_{12} compound as an α, β-unsaturated carbonyl compound with no evidence to indicate whether it was an aldehyde or a ketone. These same workers also presented presumptive evidence that lactones were responsible for the fruity and coconut

odours of certain fractions of the vacuum distillate from oxidised fat.

Fresh milk is an oxidising system with an E_h value of 0.3-0.4 volts and it has been shown by Bell[31] that the tendency for oxidised flavour to develop is directly related to the E_h value of the milk. In view of this the introduction of reducing substances into milk can be expected to delay or eliminate the development of oxidised flavour, and for this purpose the addition of ascorbic acid has been suggested by Mucha[32], Chilson, Martin and Parrish[33], Randall[34], Wright and Greenbank[35] and others.

Rôle of Ascorbic Acid

The precise rôle played by ascorbic acid in this connection is still not quite clear as Guthrie and Krukovsky[36] have shown that the complete destruction of ascorbic acid by hydrogen peroxide exerted the same protective action on the milk fat as additions of ascorbic acid, while the same authors subsequently found that flavour production was actually promoted when about half of the ascorbic acid in milk was reduced to dehydroascorbic acid. It would appear from this that over a certain E_h range the system ascorbic acid-dehydroascorbic acid can act catalytically in the transference of oxygen to fat and that this system becomes inoperative when the ascorbic acid is wholly converted to dehydroascorbic acid by oxidation or the dehydroascorbic acid is wholly converted to the reduced form by reduction.

Olsen and Brown[37,38] have shown that ascorbic acid is implicated in the development of copper-induced oxidised flavour, as they found that no oxidised flavour was produced in washed cream containing copper until ascorbic acid was also introduced. Additions of hydrogen peroxide had the same effect as the ascorbic acid under these conditions and Olson and Brown concluded from this that peroxides must be liberated during the Cu-catalysed oxidation of ascorbic acid. These workers also found that citric acid and certain amino acids exerted an inhibiting action on the Cu-ascorbic acid system and attributed this to the formation of un-ionised complexes between these compounds and Cu ions.

Guthrie[39] has shown that the removal of dissolved oxygen from milk by de-aeration before pasteurisation prevented the development of oxidised flavour, while Greenbank and Wright[40] have produced evidence that de-aeration fortifies the antioxidant activity of easily oxidisable sulphydryl compounds produced in heated milk. Krukovsky, Loosli and Whiting[41] have shown that an increase in the tocopherol content of milk fat, effected by feeding tocopherol to the cow, reduced the tendency of milk to develop oxidised flavour.

A considerable amount of work has been done on the protection of fat in milk and milk products by added antioxidants, but an effective antioxidant which is satisfactory on the basis of organoleptic tests still remains to be found. For incorporation into dairy products, the higher esters of gallic acid seem to offer the greatest possibilities in that their nontoxicity at the levels necessary to prevent the development of oxidised flavour has been established. The value of nordihydroguaiaretic acid as an antioxidant has been extensively studied, but has been considered unsatisfactory on the grounds that, at the levels at which it is effective, it gives a distinctly objectionable flavour. On the other hand, experiments by Stull, Herreid and Tracy[41a] indicate that the level at which NDGA is effective in preventing fat oxidation can be considerably reduced by simultaneous additions of citric acid, ascorbic acid or methionine, which powerfully reinforce its action.

2. *The Milk Proteins*

Recent studies of the nitrogenous constituents of milk have been concerned primarily with the fractionation of the principal protein groups, as separated from milk by isoelectric precipitation and salt fractionation, into identifiable components as indicated by their electrophoretic pattern and size distribution to the ultracentrifuge. These procedures have been applied separately to the casein fraction which separates from milk at *p*H 4.6 and the whey proteins which remain in solution after the separation of casein.

Casein.—Casein, present in milk to the extent of 3 per cent, separates spontaneously when milk is adjusted to *p*H 4.6, the isoelectric point of casein. Since Lindeström-Lang[42] showed that casein could be separated into a number of fractions, depending on their solubility in mixtures of HCl and alcohol, a considerable amount of work has been carried out with a view to separat-

ing casein into electrophoretically homogeneous fractions, to establishing their chemical identity and determining their characteristic properties.

Measurements of the electrophoretic mobility of casein in a Tiselius' apparatus by Mellander[43] showed that cows' milk casein, after separation at *p*H 4.6, consisted of three well-defined components which he designated α, β and γ caseins in order of decreasing mobility. The α and β fractions were isolated electrophoretically by Mellander and analysed. The P:N ratio of the γ-casein was found to be larger, and of the β-casein smaller, than that of the total casein.

Occurrence of Proteolysis

Warner[44] subsequently showed that γ-casein had a lower iso-electric point than β-casein and that separation of the two could be effected by the partial precipitation of casein from milk by approaching its iso-electric point from either side. He further showed that as casein contains a proteolytic enzyme, all preparations of caseins except those which have been exposed to heat or extensive treatment with alcohol, are slowly hydrolysed with the production of a large number of non-dialyzable fragments which are electrophoretically distinct.

More recently, Cherbuliez and Baudet[45] following on the previous work of Cherbuliez and Jeannerat[46], have isolated four distinct fractions from casein which they have designated α, β, γ and σ caseins. The α, β and γ caseins apparently correspond to those separated electrophoretically by Mellander while the fourth fraction, the σ casein, which remains in solution when milk is rennetted, is probably identical with Hammersten's proteose. The percentage composition of casein found by Cherbuliez and Baudet is as follows:

Component	Iso-electric point	Percentage of total casein
α	*p*H 4.5	60
β	*p*H 4.9	25
γ	*p*H 5.2	10
σ	—	5

By differential solubility in 5 per cent ammonium sulphate at *p*H 6.0 α-casein was further separated into two fractions which were designated α_I and α_{II}. These workers suggest that the α-caseins are made up of α_I and α_{II} paracaseins[47]. α_I casein containing the two paracaseins in equal amounts while α_{II} casein contains the α_I and α_{II} paracaseins in the proportion of 2:1. On the basis of this analysis of casein, Cerbuliez and Baudet have suggested an ingenious explanation for the action of rennet on milk, which is considered below.

These workers found that the Ca complexes of α and σ caseins were soluble in water over the *p*H range 5-7.5 while those of β and γ caseins were insoluble. The latter remained in solution in the presence of calcium α-caseinate, indicating that in milk the β and γ caesins are protected by the α-fraction. Electrophoretic examinations of casein solutions of varying concentrations by Nitschmann and Zurcher[48] showed that the amount of β-casein present varied inversely as the concentration of the solution. They considered this to be an indication of the formation of complexes between α and β caseins, a conclusion which supports the suggestion by Cherbuliez and Baudet that β-casein is protected by the α fraction, and the theory of Linderström-Lang[49] that the stability of the calcium caseinate system is due to the protective colloid action of one of the components.

Hipps, Groves and McMeekin[50] have separated α, β and γ-caseins by alcohol solubility and investigated the titration curves, viscosities and densities of the three fractions. Their results show that the proteins differ and that the differences in properties are related to their amino-acid composition. They also showed that preparations of the three fractions separated by alcohol or urea were the same as those obtained by the milder procedure of iso-electric precipitation at 2°, indicating that no change in structure was produced by alcohol or urea.

Amino-Acid Composition

Gordon, Semmett, Cable and Morris[51] have made a very complete examination of the amino-acid composition of whole casein, α-casein and β-casein and found significant differences between the analyses of the three proteins. α-Casein contains appreciably more alanine, arginine, lysine and aspartic acid and appreciably less valine, leucine and proline than β-casein. The differences in amino-acid composition between α- and β-casein are not in the direction that would be expected from their iso-electric points, which tends to suggest that the way in which the amino-acids are combined together differs appreciably in the two proteins.

Nitschmann and Lehmann[52] have reported that the action of rennet changes the electrophoretic pattern of casein, the α fraction being split into two parts which they have designated α_I and α_{II}. Cherbuliez and Baudet[45] and Pyne[53] have further shown that of the various casein fractions only the α fraction is attacked directly by rennet. Cherbuliez and Baudet consider that the α_I and α_{II} caseins are made up of α_I and α_{II} paracaseins in different proportions, the latter being held by bonds which are broken by rennet to expose phosphoric acid groups, attached to serine, which were masked in the untreated casein.

The exposure of the phosphoric acid groups results in an increase in the base-binding capacity displayed by paracasein compared with the intact casein, and also for the increased sensitivity of paracasein to calcium ions. The coagulation of the α-fraction by rennet results in the loss of the protecting colloid layer by the β- and γ-caseins and as these fractions are insoluble in the presence of calcium ions, they separate spontaneously with the paracasein derived from the α fraction. The soluble σ-component, which is unaffected by rennet, passes into the whey.

The effect of temperature on rennet coagulation, originally investigated by Berridge[54] has been further investigated by Pyne[53] who has shown that while rennet coagulates α-casein at temperatures down to 0° in the presence of relatively low concentration of calcium ions, β-casein is unaffected and remains in solution at temperatures below about 20°. β-casein is thermolabile and precipitates above 20° but redissolves on cooling. The fact that rennet does not coagulate milk below 20° is considered by Pyne to be due to the protecting action of β-casein on the enzyme-altered α-casein, and only when the β-casein is itself heat-coagulated is the enzyme-altered α-casein brought under the influence of calcium ions which cause it to coagulate.

Chevalier, Mocquot, Alais and Bonnat[55] have reported that variations in rennet-coagulating time are bound up with differences in the make-up of the casein molecule or complex. The caseinate obtained by supercentrifuging slow-clotting milk was found to have a lower Ca : N ratio, a smaller particle size and a greater degree of hydration than that of fast-clotting milk.

The Whey Proteins.—Whey contains about 4-5 gm. per litre of protein made up principally of immune lactoglobulins, β-lactoglobulin and a small amount of albumin and other unidentified constituents. The constituents of the whey proteins have been separated electrophoretically by Smith[56] who found the following:

	Immune lactoglobulins		Components			Component
	Euglobulin	Pseudoglobulin	a	b	β-lactoglobulin	c
Concentration ..	6	4	16	9	58	7

The colostral and milk globulins which are associated with immunity to disease are called '*immune lactoglobulins*' although it is realised that the actual antibody content may account for only a small portion of this fraction. Together they constitute 10 per cent of the total protein of the whey but the amount can be increased by hyper-immunisation. The immune lactoglobulins of colostrum and milk are apparently identical and possess molecular weights of the order of 180,000; they are separated from whey by half-saturation with sodium sulphate at *p*H 5.8. Dialysis at the iso-electric point (*p*H 5.85) causes a separation of this fraction into euglobulin (insoluble) and pseudoglobulin (soluble).

β-Lactoglobulin, first described by Palmer[57], is obtained from the filtrate from the separation of the immune lactoglobulins by saturating with sodium sulphate at 30°. The precipitate is dissolved in water, dialysed at 0° for 12 days against distilled water, the solution adjusted to *p*H 5.2 and allowed to crystallise. The crystals are insoluble in water at *p*H 4.5-5.5 but are readily soluble in dilute salt solutions. About 60 per cent of the total whey protein is β-lactoglobulin.

Recent studies of the electrophoretic pattern of β-lactoglobulin by Li[58] have indicated that it is not homogeneous; but this is denied by Bosshardt, Moore and Brand[59] who suggest that the apparent heterogeneity of β-lactoglobulin is produced by recrystallisation. Due to the relative ease with which β-lactoglobulin can be prepared in the crystalline state it has become one of the most intensely studied of proteins.

Brand, Saidel, Goldwater, Kassall and Ryan[60] have made a complete amino-acid analysis of the protein and compared it with casein. The minimum molecular weight calculated from the amino-acid analysis was found to be 42,000 and as this is of the same order of magnitude as the molecular weight determined by other procedures, the protein can be regarded as mono-molecular. The protein was shown to consist of 370 amino-acid residues united by 366 peptide bonds and arranged in four dissimilar polypeptide chains. Linkages other than peptide linkages were present only in small number.

The molecular weight of β-lactoglobulin calculated from X-ray diffraction data determined on the wet crystals was found to be 35,400[61] and from sedimentation and diffusion constants to be 35,400[62]; molecular weight determinations based on osmetic pressure measurements gave values of 38,000[63] and 35,050[64], while from monolayers spread on concentrated salt solutions the value 34,300 was obtained[65].

Briggs and Hull[66] have studied the heat denaturation of β-lactoglobulin and found that the process proceeded in two stages. The primary process, initiated at temperatures above 65°, was not accompanied by a change in electrophoretic mobility but was accompanied by an approximately fourfold increase in particle weight. The secondary process, occurring at a maximum rate at 75-80°, took place only after the first had occurred and resulted in an increase in the electrophoretic mobility. Further studies on the subject by Larson and Jenness[67], based on the activation and oxidation of sulphydryl groups, have shown that the primary process follows first-order kinetics and has an energy of activation of about 80,000 cal. per molecule over the temperature range 64-75°.

Whey Albumin

Polis, Shmukler and Custer[68] have shown that 3-5 per cent of the total protein of whey has the same electrophoretic mobility as blood serum albumin. After removal of β-lactoglobulin from whey with saturated ammonium sulphate, they precipitated the albumin with ammonium sulphate containing borate and alcohol and crystallised it from alkaline ammonium sulphate in the presence of phosphate and caprylate ions; 0.5-1.0 gm. of crystalline protein was obtained from 15 gallons of skim milk. The properties of this whey albumin are compared with those of blood serum albumin in the following table:

	Serum albumin	Whey albumin
Percentage nitrogen	16.0	16.1
Molecular weight	69,000	69,000
Optical rotation*		
α^{25}_{546}	-78 ± 2	-75
Iso-electric point	4.71	4.72
E $^{1\text{ per cent}}_{1\text{ cm.}}$ at 280 mμ	6.6	6.6

* Measured in acetate buffer of ionic strength 0.1 at *p*H 4.8.

The constituents of approximately 25 per cent of the whey proteins have not, so far, been positively identified. Sorensen and Sorensen[69] have separated a lactomucin, a green mucoid fraction, a red fraction and a crystalline protein insoluble in water and dilute salt solution, but what relationship these fractions bear to the unidentified components which have been shown by electrophoresis is to be present in whey, is not known.

THE EFFECT OF HEAT ON MILK

Studies on the effect of heat on milk have been concerned mainly with the production of heat-induced flavours, the effect of heat on the stability of the milk proteins and heat-induced interactions between certain of the milk constituents.

The so-called 'cooked' flavour of milk increases with increasing heat treatment. Gould and Sommer[70] have shown that up to 95° the increase in flavour runs parallel with the production of sulphydryl, indicating that protein denaturation is mainly responsible for the flavour developing over this temperature range. Hutton and Patton[71] have shown that the sulphydryl is derived from denatured lactoglobulin.

Above 95° the flavour due to sulphydryl is displaced in favour of a 'burnt' or 'caramel' flavour. Hankinson, Ellertson and Gleim[72] have shown that this burnt flavour component and its precursor are both dialysable, but attempts to isolate the burnt flavour component were unsuccessful due to its labile nature; it was slowly inactivated or destroyed by all the procedures employed to concentrate it. With more drastic heat treatment, browning of milk occurs with the production of an astringent flavour and a more strongly pronounced caramel flavour (sometime called 'rice-pudding' flavour). The compounds responsible for these flavours have not been identified

but the astringent flavour arises probably from heat-induced acidity and the caramel flavour from casein-lactose interaction. Patton and Josephson[75] have shown that the flavour does not develop in heated whey, indicating that the whey proteins are not involved.

Heat-induced acidity arises from the decomposition of lactose with the production of formic acid[74], lactic acid [75] and also by an increase in the acidity of casein due to elimination of basic NH_2 groups through their interaction with lactose[76]. Other compounds produced by the heat decomposition of lactose in the presence of protein in autoclaved milk are furfuryl alcohol, 5-hydroxymethylfurfuryl[77], maltol[78], acetol, acetaldehyde and acetic acid[79].

Combination of Protein & Lactose

The heat-induced combination of lactose and protein in milk has been extensively studied and is receiving further attention in view of its influence on the heat stability of casein in milk and the biological value of the milk proteins. Grimbleby[76] has shown that the combination of lactose and protein in milk proceeds in two well defined stages.

In the first stage, proceeding at temperatures up to 80°, loosely combined complexes form from which the lactose can be recovered by treatment with a strong acid. In the second stage, which proceeds at temperatures above 80° and only after the first stage is complete, lactose and protein combine to form stable compounds from which the lactose cannot be released. Further prolonged heating beyond this stage results in the formation of brown ('humin') compounds whose composition has not so far been determined. The precise mechanism by which lactose-protein complexes are converted to humin compounds is not known.

Kass and Palmer[80] have suggested that the browning of autoclaved milk can be accounted for on the basis of the caramelisation of lactose by casein and the absorption of lacto-caramel by the colloidal caseinate. Lewis, Esselen and Fellers[81] have concluded that in the browning of foods a reaction between reducing sugar and N-free carboxylic acids played an important rôle. On the other hand Mohammed, Fraenkel, Conrat and Olcott[82] have shown that the primary site of the browning reaction was the free amino groups of protein which decreased as the reaction proceeded and that masking of these groups by acetylation prevented browning at relatively low temperatures. At higher temperatures (70° and above) secondary reactions possibly due to guanidyl and indole groups were involved.

Patton, Hill and Foreman[83] and Lowry and Thiessen[84] have shown that the protein groups implicated in the combination of aldose sugar and casein are the free amino groups of lysine, arginine and tryptophane and that the loss of these essential amino-acids results in a considerable fall in the biological value of the protein. The reaction between proteins and sugars in the dry state has been investigated by Lea and Hannan[85] who also found that these basic amino-acids were immediately involved and that methionine and histidine were also lost when casein and glucose were stored together under conditions of high humidity.

The factors governing the heat stability of milk are, for the most part, unknown. The stabilising affect on the milk proteins of high temperature preheating and the addition of stabilising salts has been considered in the past to be due to their influence on the concentration of soluble calcium, but evidence is accumulating which suggests that lactose-protein complexes are, in fact, involved.

Evaporated Milk

Experiments by Deysher, Webb and Holm[86] have shown that milks of normal composition produced evaporated milks of least stability when preheated at temperatures below 75°, and that the stability of the evaporated product increased with increase in pre-heating temperature above 75°. This finding, considered with that of Grimbleby[76] that stable lactose-protein complexes are not formed in milk below 80°, suggests that lactose-protein combination is a factor which contributes to the heat stability of milk.

Bell and Webb[87] have shown that the stability of evaporated milk paralleled the production of brown colour produced during fore-warming and Pyne[88] has shown that the coagulation time of milk held at 130° increased with decreasing *p*H value of the coagulated milk, i.e., with the amount of heat induced acidity which has been shown by Grimbleby[76] to be due in part to lactose-protein interaction. Further, Mohammed *et al.*[82] have shown that a glucose-albumin product formed by heating blood serum albumin and glucose together in a buffered sub-

strate resisted coagulation by heat at 100° for several hours. The mechanism by which sugar-protein complexes exert their stabilising effect is, at the moment, open to conjecture, but the fact that they are themselves heat-stable and powerfully lyophilic is probably of importance.

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Copper Ore Process

A FLUO-SOLIDS roasting process for copper ore is being tested at Melbourne by the Australian Commonwealth Scientific & Industrial Research Organisation by arrangement with the Mount Lyell Mining & Railway and Mount Morgan companies. The copper is extracted from the roasted product by leaching and electrolysis.

Announcing this, Mr. W. E. Bassett, the Mount Lyell chairman, said there was no immediate prospect of any major change in the company's metallurgical processes at Queenstown, but the new process appeared to offer great savings in cost.

Licences for Sulphur Imports

FOLLOWING the recent announcement by the Ministry of Materials that private trading in sulphur and iron pyrites would be resumed on 1 January, 1954 (see THE CHEMICAL AGE, **69**, 1284), the Board of Trade announce that they are now prepared to consider applications for licences to import sulphur from any source from that date.

Applications for licences should be made on Form ILB/A (Revised) and should be addressed to Import Licensing Branch, Board of Trade, 43 Marsham Street, London, S.W.1. The form should be fully completed, and in particular the country of origin, the price, the source, and, if possible, the firm or firms who are to use the imported sulphur and whether it is to be used for making sulphuric acid or not, should be stated.

Imports of iron pyrites from any source are now admissible under Open General Licence and separate import licences are no longer required.

Copper & Cobalt in Uganda

REGARDED as the most important mining project in Western Uganda, the Kilembe copper and cobalt mine is expected to come into production in 1956 as a result of an agreement whereby the necessary finance will be provided by Frobisher Ltd. (the Canadian mining concern and subsidiary of Ventures Ltd.), the Colonial Development Corporation and the Uganda Development Corporation.

Total expenditure to complete the scheme is estimated at £6,500,000. Frobisher Ltd. have already spent about £2,000,000 on the project and now the Colonial and Uganda Development Corporations have agreed to subscribe £2,500,000, Frobisher Ltd. to provide the balance.

The initial output of the mine when it comes into production has been estimated at 40,000 tons of ore a month, from which will be produced a copper concentrate and a cobalt pyrite concentrate. Some 18,000,000 lb. of copper and 900,000 lb. of cobalt may be produced annually.

The new mine, which will be served by the new railway now being built between Kampala and Kaseve on Lake George, is considered to be a major step forward in Uganda's economic development. It has been estimated that it will provide employment for about 1,300 Africans and Asians, as well as more than 100 Europeans.

Dechema Institute Opened

Apparatus & Materials to be Listed

ON 4 Decmber the Dechema Institut für Apparate und Stoffkunde (Dechema Institute for Chemical Apparatus and Materials) was formally opened in Frankfurt-am-Main. Some 200 well-known personalities in scientific and industrial circles, including a number of foreign visitors, were present at this function, when Dr. Hans Broche, of Essen, and Senator E.h. Dr. Bretschneider, chairman and managing director of the Dechema respectively, made some illuminating remarks concerning the aims and objects of the Institute.

The Institute represents an attempt to illustrate in a practical manner the broad area covered by the field of chemical apparatus and equipment. Prof. Dr. Grassmann of Zurich reported on the success that he had with a series of lectures, which ran through several terms, on the subject of chemical apparatus, equipment and materials.

The Dechema Institute will classify in one register (the Dechema Register) all chemical apparatus and equipment with respect to its theoretical bases of design, its construction, mode of operation and application to industry. A compilation of 100 materials with full particulars of their physical properties and resistance to 1,000 active chemical agents is now appearing in a third edition. Dechema-Literature-Schnelldienst (Dechema Review and Reporting Service) also forms a part of the activities of the Dechema Institute.

The demonstration and display halls of the Institute will enable visitors, both German and foreign, to obtain a broad bird's-eye view of the present position of the chemical apparatus and equipment field. At the same time, visitors will also be afforded an opportunity of operating and testing the equipment and apparatus themselves. It is also intended that discussion groups shall be formed in connection with the demonstrations. Plans are also being formulated whereby German foreign students will be able to take part in a series of lectures and educational group meetings to be held during the University vacations.

Progress in Permeable Porous Ceramics

by **FELIX SINGER, Dr.Phil., Dr.-Ing., M.I.Chem.E.**

CERAMICS is a term covering all those products made of inorganic materials which are first shaped and then hardened irreversibly by heat. The term 'porous ceramics' in the general sense is self-explanatory.

Many traditional ceramic products are porous, e.g. bricks, tiles and earthenware. This is incidental to their method of production and not necessarily desirable. In order to make them impervious a glaze must be applied, as in earthenware crockery, wall tiles, etc.

I want to consider here a field of ceramics where the porosity is an essential feature of the ware. It is concerned with the passage of fluids through ceramic pieces for various purposes, and has been developed for filtration since the beginning of the century but now has wider applications. I propose to call it 'permeable porous ceramics.'

Progress in the development of permeable porous ceramics of known high chemical, thermal and mechanical resistance, definite pore size, and maximum permeability, in various shapes and sizes, was greatly accelerated by wartime needs.

They are made from a single, pure, raw material of high chemical resistance. For normal use it could be silica sand, fireclay or porcelain grog, kieselguhr (diatomaceous earth), corundum, carbon or composite refractory ceramic bodies; for especially high mechanical strength and resistance to abrasion and heat, corundum or silicon carbide is used.

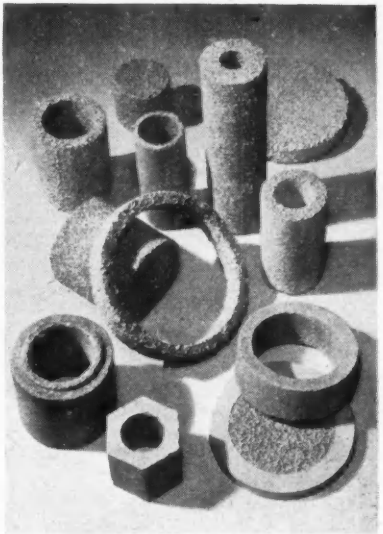
The raw material is size graded, and particles of one particular size only used for any one product. They are bonded with the minimum of flux. In this way articles with uniform pore distribution and a porosity of at least 25 per cent are produced. The maximum pore size of any particular grade is kept within limits suitable to the grade; for instance, a coarse industrial grade may vary from 650-750 μ whereas fine grades are very uniform and adjacent pores are classified with maximum pore sizes of 2.5 μ and 2.0 μ respectively.

A number of sizes of tubes with both ends or one end open, and a variety of round or rectangular tiles are in general production. Some grades can also be obtained with non-porous surrounds, the porous and nonporous part being fired together[4]. These are very useful when filtering corrosive chemicals which attack the cements otherwise used to fix filtering plates. (Fig. 1.)

TESTING

The British Standard[1] for testing these materials describes the measurement of maximum pore diameter, and of absolute permeability. The first can be carried out rapidly for checking purposes; the second is a more lengthy process and is not applied in routine checking.

The maximum pore diameter is measured by finding the minimum pressure of air required to force a bubble through the filter. The pores in the test piece are first filled with the test liquid (usually water, for very fine pores carbon tetrachloride or amyl acetate) the piece is then submerged in the liquid and air pressure is gradually applied



[*Photograph by courtesy of Royal Doulton Potteries*

Fig. 1. Some industrial porous ceramic articles

to one side until the first bubble breaks away. The calculation is based on the passage of an air bubble through a capillary tube of circular cross section:

$$D = \frac{4 \times 10^3 \alpha}{981 (P - g\rho h)}$$

where D = maximum pore diameter in microns
P = bubble pressure recorded, in millimetres of water.
h = depth (in mm.) below the liquid surface of the point at which the bubble is first observed (can usually be neglected)
α = surface tension of liquid in dynes per cm.
ρ = density of liquid.

Expressing the pressure P in millimetres of mercury the equation can be simplified to:

$$D = \frac{30 \alpha}{P}$$

Some measure of uniformity of pore diameter can be demonstrated by increasing the pressure by a small amount after the first bubble has formed, but this method must be employed with circumspection.

The permeability is found by measuring the rate of flow of a fluid (water) through the test piece when a known pressure difference is maintained across it.

$$\phi = \frac{V\ l\ \eta}{981\ Apt}$$

ϕ = absolute permeability
V = volume in ml. of fluid in time t seconds
l = thickness of filter in cm.
η = viscosity in centipoises of permeating fluid
A = effective area of filter in sq. cm.
p = pressure difference across filter in cm. of water.

Photograph by courtesy of Royal Doulton Potteries

Fig. 2. Large size porous tube for dust recovery

USES OF POROUS CERAMICS

1. *Filtering.* Porous ceramics have been used for filtering in the chemical industry for at least fifty years. They have the advantage over paper or cloth filters of being chemically inert, of withstanding heat (up to 1,000°) and of being rigid. Compared with sand or diatomaceous earth filters which share the same chemical and thermal inertness, the rigidity of porous ceramics gives the advantage that they can be used in any position.

Being rigid and inert a ceramic filter can be efficiently cleaned by reversing the flow or by chemical means. When put back into use they do not require a settling down period, like cloth filters, and, when used with a filter aid, they do not need precoating.

There are many uses for porous ceramic filters in the chemical industry. Solid material of sizes down to almost colloidal dimensions can be filtered out from most liquids, although very viscous oils with a low solid content do cause difficulties.

The industrial grades produced by one of Britain's leading firms in this field are listed as follows[3]:—

Measured Maximum Pore Sized in Microns	
650-750	approx.
300-350	,,
150-175	,,
90-110	,,
75-90	,,
50	,,
35	,,
25	,,

They will usually retain solid particles down to a size approximately one-third of that of the measured maximum pore size. They have a high permeability so that the flow-rates of filtering systems using them

depend more on the nature and resistance of the deposit built up on the filter and the restrictions in the flow of the whole system than on the filter itself.

Filtering units may be built up from flat porous plates, etc., and a number of types of ready made filtering units containing candles are available for laboratory, pilot plant and industrial uses. Pipe line filter units containing a filter element 18 in. long and 3½ in. outside diameter have an effective surface of about 1¼ sq. ft. The element is encased in a hard glass tube of approximately 4¼ in. bore and has chemical stoneware inlet and outlet connections at either end.

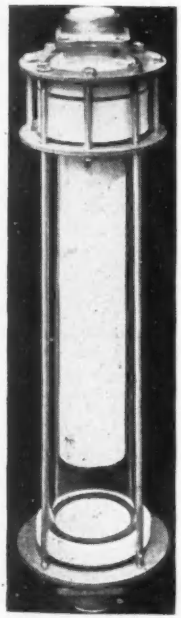


Fig. 3

The glass casing can be removed for cleaning purposes without disturbing the filter element and thus contaminating the filtrate. (Fig. 3.) Various pressure filter units are encased in stainless steel or enamelled cast iron and contain one or more candle elements. There are also completely metal free, acid resistant chemical stoneware vacuum filter units of up to 3½ gal. capacity.

The filtering of drinking water through kieselguhr Pasteur filter candles is well established. During the recent war similar methods were adapted for larger mobile filtration units for the forces. They proved to save space, weight and time when compared with the old sand method, and, as they do not need precoagulation, they need not be worked by skilled men[8].

A more recent development of this type of filtration is its successful application to the sterilisation of liquids that may not be heated.

In 1950 Royce and Sykes[9] published work done in the Boots Microbiology division on effective filtration of bacteria from fair quantities of liquid over a period of several hours. As a measure of pore size they used the 'bubble-pressure' test and found that reliable filtration over a period of six to seven hours can be achieved with Kieselguhr or other fine grade candles with 17 psi. bubble pressure.

Candles with this bubble-pressure are equally efficient whether they are thin or thick walled. Lower pressure candles often give bacteria-free filtrates for short periods but breakdown occurs after a certain time. Thicker walled low pressure candles retard the passage of the bacteria but do not prevent it. Moreover, 17 psi. candles stand up to sterilisation, whereas the lower pressure ones do not always. The satisfactory sterilisation of liquids is of considerable importance in the pharmaceutical industry.

Porous ceramics find considerable use in the filtration of gases in ducting and pipeline systems, cyclones, etc. They replace canvas bags, which lack the rigidity and chemical and thermal inertness of the ceramics, and hence need a settling down period after each cleaning. Also, a minor blemish on a canvas filter perforates it and easily becomes a tear whereas a slight injury only affects the surface of the ceramic. Cleaning of ceramic gas filters is simply by blow-back which can be made automatic; and they show no marked deterioration over periods to 12 to 18 months' constant use. (Fig. 2.)

Small porous ceramic plugs have recently been introduced to avoid spilling the mercury from manometers. They are made ¼ in. in diameter and ⅜ in. long, and are intended for use in the end of the manometer open to the atmosphere. The grade of material is so chosen that there is no appreciable delay in reading the manometer. But if too high pressure is applied to the manometer, so that the mercury would normally overflow from the open end, the porous plug will hold it back up to an excess pressure of 11 cm. of mercury.

2. *Diffusion.* Porous ceramics are used for aeration and diffusion. They are applicable where gases have to be dissolved in liquids or so intimately mixed as to react with them. In other instances an inert gas, usually air, is passed to accelerate mixing and chemical reaction. Diffusers are also used to blend one liquid with another.

Direct applications are in the activated sludge method of sewage treatment, in the cyanide process of treating ore slimes, or in

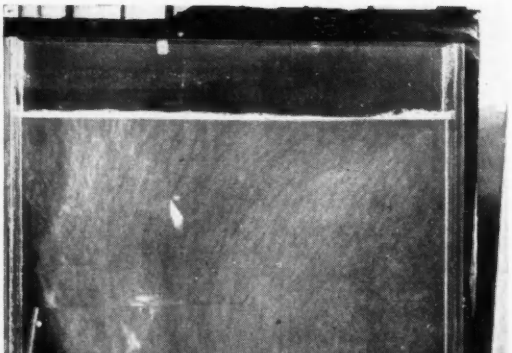


Fig. 4. Gas diffusion candle in a tank of water, showing the even bubbling obtained

[*Photograph by courtesy of Royal Doulton Potteries*

froth flotation cells for ore concentration. Sometimes the same unit can be used for aeration and filtration as in the aeration of water to precipitate the dissolved iron (which is then filtered off) or the separation of solids in some chlorination processes[6].

The most usual application of ceramic diffusers is, then, for passing gases into liquids. Candles or fitted plates are generally used. (Fig. 4.) With ceramic diffusers, bubbles of smaller diameter and more even distribution can be achieved than with other means. The minimum pressure required to produce bubbles depends on the hydrostatic head of liquid above the diffuser, the resistance of the materials to fluid flow and the capillary action of the liquid in the pores of the diffuser. This last factor is large and necessitates equipment that will supply the gas at relatively high pressures.

Also, since the pores of a ceramic diffuser are not completely uniform in size, the necessary pressure to force the gas through the smaller ones is not reached and these become redundant. If solids, either crystalline or growing organisms, form in the aerated liquid they can clog up the liquid filled pores and may even grow into the larger ones and stop the gas flow.

The recent innovation[5] for making the diffuser unwettable is therefore of considerable importance. It is achieved by coating the whole surface with a silicone. The capillary action then ceases and the pores only fill with liquid under hydrostatic pressure. Application of gas pressure to one side will clear all the pores of the liquid when the pressure is sufficient only to overcome the hydrostatic pressure and the flow resistance. The gas bubbles are somewhat larger than those from an untreated diffuser of the same grade.

Air diffuser tiles have recently aroused interest in their application to the fluidisation of powdered materials. It is well known that powdered or granular material when poured on to a flat surface will form a cone with a definite angle of repose dependent on interparticle and particle-surface friction. Because of this, powders on an incline do not move until the slope is greater than the angle of repose. Transport of powders by chutes therefore necessitates a steep incline, and if the distance is large the elevation at the beginning may need to be uneconomically high. If, however, the powder rests on a porous surface through which air is passing the particles are partly lifted up, the interparticle friction becomes reduced to a minimum and the angle of repose is destroyed.

If the base of the chute is similarly porous and supplied with air, powders can be made to flow along it at slopes of less than 5° to the horizontal. This is called an 'air slide.' It consists of a trough which may, or may not be closed at the top, with a layer of closely fitting porous tiles some small distance from the bottom cutting off a lower compartment. This is supplied with an inlet for filtered air, either compressed, or drawn in by suction in the upper compartment. The powder is conveyed along the top of the tiles. (Fig. 5.) So far the air slide has only been applied in comparatively few industries of which the cement industry is the most important but experiments have shown its suitability for numerous materials[2]. There is no apparent reason why it cannot be used very successfully for numerous other products, and by using hot or cold air, drying and heating or cooling could be executed en route[1].

Diffused air also assists greatly in drawing off powders from hoppers where they otherwise bridge and cake. Porous tiles are fitted in the sides of the hopper and air is blown in.

3. *Absorption.* The fine pores and high porosity of porous ceramics make them very suitable for absorbing liquids. Tiles are used for drying crystals, etc., where choice of the right grade assists in giving the best results. They have recently been adopted in a new photographic process for the reproduction of printed matter which requires wiping over the print with chemicals. As it is undesirable for the surplus chemical from one print to get on to the underside of the next the use of a porous tile to lay the prints on for treatment presents a constantly dry surface.

The absorptive powers of porous ceramics also make them a good slow source of liquid, and hence, their use as seed saucers for germination of seeds under test, and for conveyance of liquids required to evaporate slowly.

4. *Electrolytic Diaphragms.*—Permeable porous ceramics are also used to perform duties directly opposite to those of diffusers. They are employed as diaphragms in electrolytic cells to keep the different liquids apart, while offering low resistance to the passage of current. This well established use led to the development of a device for lengthening the period of service of chromium plating solutions, during the shortages of the recent war. The application of this 'dechromator' is now fairly widespread.

During normal chromium plating, the solution, which, to begin with, contains hexavalent and trivalent chromium in suitable proportions, is oxidised at the anode and reduced at the cathode at different rates and gradually gets too high a trivalent content. The trivalent chromium combines with the hexavalent, to give a compound of low conductivity.

The dechromator consists of a porous pot about 18 in. deep and 6½ in. outside diameter, fitted with steel electrodes fixed to a hook. It is hung from the cathode bar and a heavy current passed. Oxidation at the anode occurs normally, but the cathode reduction is prevented; so that the proportion of hexavalent chromium is increased. By using it at suitable intervals the correct proportions of the two valencies of the chromium can be maintained in the bath[3].

The ceramic industry is now able to supply permeable porous ceramic ware of definite pore size and high permeability in a

[*Photograph by courtesy of Royal Doulton Potteries*

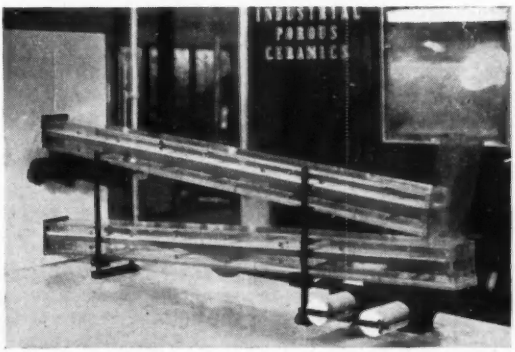


Fig. 5. Model illustrating the principle of a static-bed pneumatic conveyor

large number of shapes and sizes. Their applications have become more varied in the last ten to fifteen years and it seems likely that industry will find many more processes where porous ceramics are of assistance.

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Plastic Material Analysis

Some Physical Methods Employed

THE ninth annual general meeting of the Physical Methods Group of the Society of Public Analysts and Other Analytical Chemists was held at 6 p.m. on Tuesday, 24 November, 1953, in the Meeting Room of the Chemical Society, Burlington House, Piccadilly, London, W.1. The chair was taken by Dr. J. Haslam, F.R.I.C.

The following were elected officers of the Group for the ensuing year: *Chairman*, A. A. Smales, B.Sc., F.R.I.C.; *vice-chairman*, J. E. Page, B.Sc., Ph.D., F.R.I.C.; *hon. secretary and treasurer*, R. A. C. Isbell, A.Inst.P., Hilger & Watts Ltd., Hilger Division, 98 St. Pancras Way, London, N.W.1.

The annual general meeting was followed by the forty-second ordinary meeting of the group at which the new chairman, Mr. A. A. Smales, took the chair, and about 55 members and visitors were present. The retiring chairman, Dr. J. Haslam, gave an address on 'Physical Methods in the Analysis of Plastic Materials—Some Observations of an Analytical Chemist.'

Dr. Haslam said that physical methods were largely used in the plastics industry; but although some of them were purely physical, many of the tests used required preliminary chemical separations before a physical method could be applied. Of the purely physical methods, the determination of relative viscosity at a specified concentration in a suitable solvent was in regular routine use.

Potentiometric titration had supplied information on the speed of hydrolysis of Nylon, polymethyl methacrylate and polyvinyl chloride, that was difficult to get by chemical means. Chromatography had yielded valuable results in the examination of Nylon polymers, as had partition between immiscible solvents in the analysis of their hydrolysis products. Spectrographic methods were largely used in the examination of plastics and their constituents after chemical or physical separation. The infra-red was the most used region, in the analysis of Nylon and polyvinyl chloride, and for simple and mixed plasticisers it often led to results not otherwise obtainable.

The visible region was used in the identification of dyestuffs; and the ultra-violet in the determination of the efficiency of UV absorbers in 'Perspex' sheet; in the detection of catechol in Nylon; of acenaphthalene in its co-polymers; and, in conjunction with a fluorescent screen, for the detection of butyl phthalate in 'Perspex.' The carbon arc was useful in the detection of inorganic fillers.

The refractive index was of importance in the examination of monomers and plasticisers.

Amperometric titration had been used, with a rotating platinum indicator electrode and a reference half cell containing potassium iodide-mercuric iodide and potassium chloride solution, for the determination of lauryl mercaptan by means of standard silver nitrate solution.

Polarograph Used

The polarograph had been used in the determination of phthalate esters in the presence of castor oil, which invalidates the fluorescein and phenol-phthalein tests; as also for the determination of zinc in fillers.

The method of partition between immiscible solvents had enabled fatty acids from the hydrolysis products to be separated and determined.

In plastics containing fillers the microscope was indispensable and in conjunction with Wallace's lycopodium method it had been used for the determination of coconut-shell flour, used in phenol formaldehyde moulding powder to give a polished finish to the goods.

Organic Chemistry : Naturally-Derived Compounds

by J. R. MAJER, M.Sc., Ph.D., A.R.I.C., D.I.C.

THE most interesting feature of the investigations in organic chemistry reported during the last year has not been the concentration of attention upon the isolation, identification and synthesis of natural products, but rather the tendency to use 'natural procedures' for carrying out these operations. The utilisation of enzyme systems either in the living microorganism or as extracts from the organs of higher animals to carry out chemical reactions is far from being a novel idea, but the incorporation of these biochemical techniques into general synthetic and analytical organic chemistry is comparatively new.

The older workers were always faced with the problem of extracting their products from the culture broth or the enzyme preparation, a formidable task which has now been simplified by the development of the chromatographic method and by ion exchange procedure. The advantages of using enzymes are many, since the reaction may be carried out at low temperatures and the yields are in many cases high; moreover the enzymes are frequently specific in action and do not lead to contamination with by-products.

Microbiological Oxidation

An excellent example of this method of operation is the 'microbiological oxidation' of steroids by moulds of the *Mucorales* family discovered in 1952. (THE CHEMICAL AGE, 1953, **68**, 103.) This permits the direct introduction of a hydroxyl group into the steroid molecule by adding the substance to a growing culture of the mould. The product is isolated after a suitable lapse of time.

Two species of moulds have been examined, *Rhizopus nigricans*, which is capable of introducing a hydroxyl group into the 11α position and *Rhizopus arrhizus*, which favours instead the 6β position. Among the examples which have been quoted are the oxidation of 17α-hydroxy progesterone to both 11α- and 6β-17α-dihydroxy-progesterones, the conversion of 11-desoxy-corticosterone to both epicorticosterone and 6β-hydroxy-11-desoxy-corticosterone. *R. nigricans* has also been used to convert 6-dehydro-progesterone into 11α-hydroxy-6-dehydro-progesterone and to produce the 11α-hydroxy derivatives of pregnan-3,20-dione and *allo*-pregnan-3,20-dione.

Facilitates Syntheses

Since one of the most difficult steps in the production of cortisone from other naturally occurring steroids is the introduction of an 11 oxygen function, microbiological oxidation facilitates a number of partial syntheses. Thus 11α-hydroxy-progesterone has been transformed into cortisone by a ten-stage synthesis involving hydrogenation to 11α-hydroxy-pregnan-3,20-dione followed by oxidation to the trione and selective reduction at 3 with sodium borohydride.

Reichstein's compound S has also been converted to the 11 epimer of compound F which is readily transformed in good yield into cortisone acetate. Other 11α-hydroxy analogues of 11β-hydroxy biologically important components of the adrenal cortex such as 11β-21-dihydroxy-progesterone have been prepared.

There has been a great deal of investigational work with conventional procedures on cortisone intermediates and steroid chemistry generally. As an example, one such route involves the isomerisation of 9α,11α-epoxy compounds to the corresponding 11α-ketosteroid with boron trifluoride-ether as catalyst. The formal total synthesis of epiandrosterone has also been reported, the starting material being tetrahydro-1,7-diketo-2,13-dimethyl phenanthrene. Other members of the steroid group to which this synthesis applies are testosterone and vitamin D_3 as well as cortisone.

The difficulties of the cortisone synthesis have prompted the study of simple analogues of the steroid in which the side chain is attached to more accessible nuclei. Examples of these are 1-glycollyl-cyclohexanol and 1-glycollyl-2-methyl-cyclohexanol.

Studies in the purification of the adrenocorticotropic hormone (corticotropin) have also been reported. Despite its high mole-

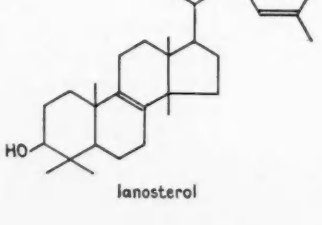
cular weight (5,000-6,000), corticotropin B, the pepsin degradation product of corticotropin, can be purified on a column of cation exchange resin, eluted with hydrochloric acid and obtained as the hydrochloride. Further purification by the countercurrent distribution technique using *sec.*-butyl alcohol and aqueous 0.5 per cent trichloroacetic acid has shown the presence of two other compounds, one of them exhibiting a low degree of activity.

One of the more unexpected discoveries is that of a new steroid with progestational activity. This activity is very specific and is confined to progesterone and its dihydro derivatives. The new compound, with an activity equal to that of progesterone, is 19-*nor*-progesterone and is produced by the 10-stage degradation of strophanthidin.

Certain alkaloids having interesting physiological properties, such as veratrine, solanine and conessine, have been shown to contain a steroid structure, and this has prompted the preparation of steroids in which a carbon atom of the skeleton has been replaced by a nitrogen atom (aza-steroids). One such compound is 15,16-dihydro-1-aza-cyclopentanophenanthrene.

ALKALOIDS

Cevine, $C_{27}H_{43}NO_8$, is another such steroidal alkaloid. It has been shown to be a tertiary alkamine containing a 'masked' secondary ketol structure, a system which is also present in the related alkamines, germine and protoverine. The structure accounts for three of the eight oxygen atoms; three more are present as non-acetylatable tertiary hydroxyl groups. One more hydroxyl group is easily acetylated and the remaining member only with difficulty. The oxidation of the alkaloid with lead tetraacetate and with periodic acid has been studied. Work upon the other steroidal alkaloids mentioned above has been reported; steroidal bases



Ianosterol

similar to the degradation products of conessine have been synthesised and the positions of the double bond and of the dimethylamino group in the conessine molecule determined.

An examination of the hypertensive alkaloids of *Veratrum album* has shown protoveratrine to be a mixture of two substances, protoveratrine A and B. The first has the structure first assigned to protoveratrine, and the second yields on hydrolysis protoverine, 2-methyl-butyric acid, 2,3-dihydroxy-2-methyl-butyric acid and two molecules of acetic acid. A third substance, germitetrine B, has been isolated from the 'amorphous' alkaloid fraction and shown to yield on hydrolysis germine, 2-methyl-butyric acid, 2,3-dihydroxy-2-methyl-butyric acid and two molecules of acetic acid.

Among the alkaloids derived from other nuclei which have been investigated are kopsine and corlumine. The second, the 3,1-isoquinolylphthalide alkaloid found in *Fumariaceae* species, has been synthesised, while the first, an extract from *Kopsia fruticosa*, has been subjected to alkaline hydrolysis. The products are kopsidine, $C_{20}H_{24}O_3N_2$, and kopsidinine, $C_{19}H_{22}O_2N_2$. Fusion with caustic potash yields 2-methylindole, indole-2-carboxylic acid and an unidentified base.

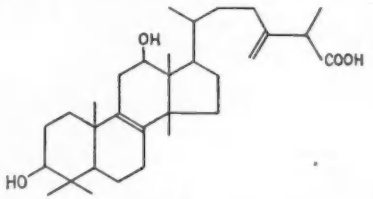
Despite the classical synthesis of morphine by Gates, alternative routes to the morphine structure continue to be suggested, one such route involving the intermediate spiroketone, 2,2'-tetramethylene-1-tetralone. Some attention has been paid to the stereochemistry of the alkaloids, particularly the tropane group, in which the configurations of the cocaines, tropine and ψ-tropine have been studied.

TRITERPENES

Most of the investigations on terpenes reported during the last year have been concerned with the relatively neglected tetracyclic group of triterpenes. The group has been subdivided into two sections upon the evidence of optical rotation and ultra-violet spectrum. Lanosterol, the triterpenoid alcohol from wool fat, and one of the most important members of the group, has been assigned a definite structure, and the rather complex stereochemistry of this structure has been examined. Because of the formal relationship of the lanosterol structure to that of the steroids, work has been initiated

to synthesise the lanosterol analogues of physiologically active steroids. The first of these series to be synthesised is the lanosterol analogue of Vitamin D_3 (7-dehydrocholesterol).

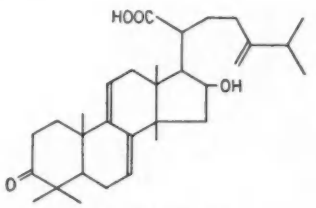
Another member of the group whose structure has been determined is polyporenic acid A, a sapogenin from the beech tree fungus *Polyporus betulinis*. It has been shown to contain two double bonds, two hydroxyl groups and a carboxylic acid group. Both hydroxyl groups are secondary but have different reactivities, as have the ketones to which they can be oxidised. On heating, the acid is smoothly decarboxylated and one of the double bonds is present in a vinylidene group, being readily hydrogenated to yield two non-interconvertible series of compounds.



polyporenic acid A

The hydrogenated acids are not readily decarboxylated, suggesting that the reactive double bond is β,γ to the carboxylic acid group in the parent molecule. The unreactivity of the second double bond suggests that it is tetra substituted. The structure of polyporenic acid A is partly revealed by oxidation of the acetates of the methyl ester. The products contain identifiable chromophoric groups which fix the position of the inert double bond and indicate a structure having close affinities with lanosterol, 3α,12α - dihydroxy-eburi - $\Delta^{8,24(28)}$-dien-26 - oic acid (see below).

A related compound from the same source, polyporenic acid C, has been characterised as a tetracyclic triterpene containing a hydroxy, a ketone and a carboxylic acid group and three double bonds. The molecular formula is $C_{31}H_{46}O_3$ and the structure was determined by conversion to a derivative of another fungus metabolic product, eburicoic acid, which in turn was degraded to a derivative of lanosterol. In fact both acids can be considered as derivatives of trimethyl ergostane.



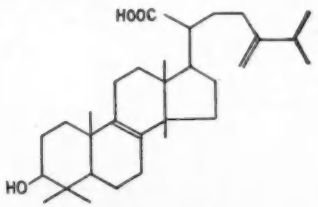
polyporenic acid C

Another interesting link between the tetracyclic triterpenes and the steroids is given by the fact that polyporenic acid C has the same carbon skeleton as the major fungus sterol ergosterol, suggesting that they may share a common metabolic route. Eburicoic acid is extracted from the fungus *S. basiomycetes*; it has been shown to have the molecular formula $C_{31}H_{50}O_3$. A related substance has been discovered in the mixed metabolic products of *Leutinus dactyloides* and characterised as a conjugated diene acid.

On oxidation of the crude mixture eburiconic acid can be isolated and shown to be identical with that prepared from eburicoic acid. This suggests that the diene acid is dehydro-eburicoic acid, and it has structural similarities to agnosterol. Eburicoic acid itself has been shown to contain three of the four rings of lanosterol with the same three angular methyl groups. The position of the hydroxyl group was fixed by cleavage to a dicarboxylic acid and pyrolysis of the di-acid to a five membered ring ketone.

The carboxylic acid group was shown to be secondary and contained in the side chain $CH(COOH)CH_2CH_2C(:CH_2)CH(CH_3)_2$. The complete structure was decided by conversion to a derivative of lanosterol, lanostan-8-ene.

Many other substances have been extracted from fungi including antibiotics and



eburicoic acid

pigments. The clear orange metabolic broth of *Chactomium indicum* yielded on extraction an acid $C_{14}H_{12}O_7$, shown to be 4-carboxy-2-oxo-3-phenyl-hept-3-en-oic acid or its lactone.

The *Aspergillus* family has received some attention. The black amorphous pigment of *A. niger*, aspergillin, has been oxidised with hydrogen peroxide to yield acetaldehyde, oxalic acid and mellitic acid. The mycelium of *A. niger* has yielded a new xanthone, asperxanthone, which has been shown to be a hydroxy-dimethoxy-methyl-xanthone. On demthylation the substance yields *nor*-rubrofusarin but so far it has not been identified with any synthetic polyhydroxy-xanthones. *A. niger* also provides a unique intercellular glucosan called 'mycodextran' or 'nigeran.' Nigeran has been shown to contain a *trans*-glucosidase responsible for the synthesis of *iso*maltose and panose from maltose and *trans*fructosidase which utilises sucrose in the formation of trisaccharides.

Another member of the family, *A. citricus*, which has the property of producing citric acid from glucose, turns the substrate a dark port wine red on disappearance of the glucose. Ether extraction of the acidified filtrate yields a red pigment, flaviolin. The pigment gives an O-trimethyl and a triacetyl derivative which can be degraded to 3,5-dihydroxy-phthalic acid, suggesting that flaviolin is 2(3),5,7-trihydroxy-1,4-naphthaquinone.

Coriolus sanguineus, a red wood rotting fungus, which can be grown on a synthetic medium, contains a red pigment, polystrictin, first isolated in 1951. It is obtained as a red crystalline solid with the molecular formula $C_{14}H_{10}O_5N_2$. Polystrictin is soluble in strong sulphuric acid and alkalies; on methylation to a mono-methyl ether it becomes soluble in dilute mineral acid and insoluble in alkali, while the acetate is soluble in alkali, but not in acid. This suggests that the hydroxyl group, proved to be *peri* to a carbonyl group with triphenyl borate reagent, is attached to a quinonoid nucleus. One nitrogen is considered to be part of an amide group while the other is heterocyclic in character, but capable of acetylation during the process of reductive acetylation to the compound $C_{20}H_{16}O_7H_2$ triacetyl-anhydro-dihydro-polystrictin.

The corresponding methyl ether did not lose water on reduction and it was assumed that a quinonoid system had been reductively acetylated and that the molecule of water lost came from the hydroxy group. Since ammonia and carbon dioxide are obtained on hydrolysis, the amide is considered to be derived from an unstable carboxylic acid. The residue of the carbon and hydrogen atoms are believed to be part of a polycyclic unsaturated structure.

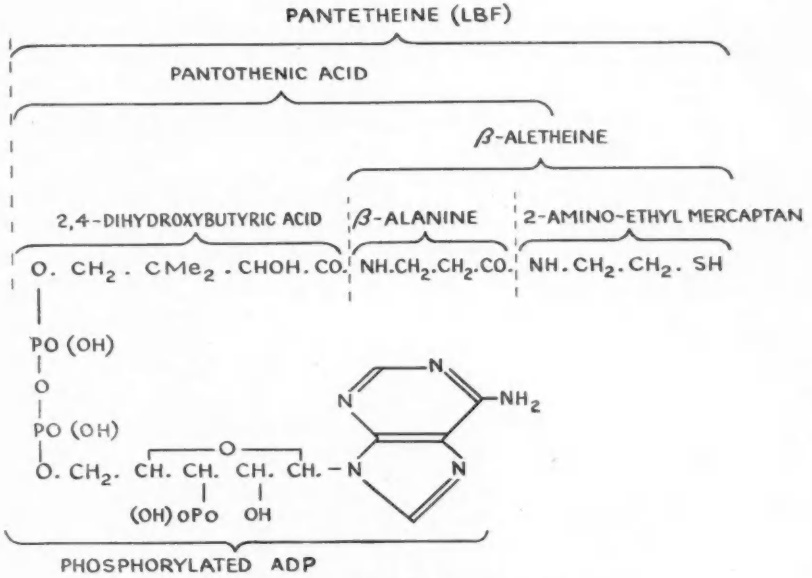
Other investigations on triterpenoids have involved phyllanthol, quinovic acid, the amyrins and butyrospermol. The last undergoes an interesting reaction in which, on addition of hydrogen chloride to the double bond of the *iso*propylidene group, there is simultaneous isomerisation of the unreactive double bond. Dehydrochlorination of the product yields an isomer of butyrospermol containing a vinylidene group. The reaction is believed to be a general one and has been applied to other triterpenes such as euphyl acetate.

PANTOTHENIC ACID, LBF & CoA

The past year has seen the elucidation of the structure of a compound of the greatest biological interest, co-enzyme A. To view this achievement in its correct perspective some of the steps leading up to it must be discussed. Pantothenic acid has been recognised for some time as a member of the B group of vitamins and its structure determined as the simple peptide of β-alanine and 2,4-dihydroxy-3,3-dimethyl-butyric acid. It behaves as an essential growth factor for some microorganisms and was postulated to be part of an enzyme system.

In 1945 co-enzyme A was recognised as an entity in the enzyme system responsible for acetylation reactions and acetyl-CoA proved to be the 'active acetate' which intervenes in many body processes such as the production of body constituents and the oxidation of food. On degradation of co-enzyme A, pantothenic acid was produced, and the former was considered to be the active form of the acid in body tissue as only very small quantities of the free acid could be detected.

Microorganisms vary in their ability to synthesise co-enzyme A; some can produce it directly from inorganic salts, some are unable to synthesise pantothenic acid and require to be fed with it, while others need an even more complex substance nearer in structure to co-enzyme A itself. Such an organism is *Lactobacillus bulgaricus* which requires for its satisfactory growth a sub-



The structure of Co-enzyme A

stance designated as LBF, which is found in most tissue and which is believed to be an intermediate between pantothenic acid and co-enzyme A.

Organisms which can manufacture CoA frequently excrete it into the surrounding medium. Thus, from the culture filtrates of *Ashbya gossypii*, LBF can be extracted. This factor is actually a mixture, separable by chromatography, of disulphides. These disulphides have been produced by the oxidation of pantetheine and inactive materials containing free-SH groups.

Acid hydrolysis of one of the mixed disulphides designated as LBF-1A gave β-alanine and the disulphide of β-mercapto-ethylamine, while enzymatic degradation yielded pantothenic acid. As LBF-1A is neutral and not destroyed by nitrous acid, the two components must be connected by an amide link and pantotheine has been shown to be identical with LBF. It has been synthesised by the condensation of methyl pantothenate with 2-amino-ethyl-mercaptan, but better yields are given by an alternative route.

Aletheine (β-alanyl-2-amino-ethyl-mercaptan) is prepared from carbobenzoxy-β-alanyl chloride with 2-amino-ethyl-mercaptan followed by reduction and isolation of the crystalline oxalate. β-Aletheine is then condensed with pantoyl-lactone to yield pantetheine of high purity and good yield. The product yielded 95 per cent of pantothenic acid on enzymatic hydrolysis and assayed 20,000 LBF units/mg.

Because pantothenic acid itself is sensitive to both acid and alkali the degradation of co-enzyme A was carried out by means of enzyme action. The enzymes used were intestinal phosphatase, and an unidentified substance extracted from liver. Both substances destroyed CoA, giving different products, but neither gave the expected yield of pantothenic acid unless used together.

Since the phosphatase hydrolysed phosphate esters, the pantothenic acid was assumed to be phosphorylated and also to be connected to a further radical by a link capable of fission by the liver enzyme. This link is identified as the amide linkage between 2-amino-ethyl-mercaptan and pantothenic acid in pantetheine. The residue of the co-enzyme A molecule must be con-

nected to the pantothenic acid residue by means of a phosphate bridge to one of the hydroxyl groups.

Co-enzyme A was now hydrolysed with barium hydroxide and the hydrolysate separated into two phosphorus-containing fractions. The first was adenosine-5′-phosphate and the second was pantothenic acid-4′-phosphate.

The question of whether these two fragments were connected by a phosphate or a pyrophosphate bridge was settled by the use of a specific enzyme, snake venom pyrophosphatase, which degraded co-enzyme A into pantetheine-4′-phosphate and adenosine diphosphate, a grouping which appears in many other substances, including co-enzymes. The position of the phosphate attachment in the adenosine was determined once again by the use of specific enzymes which showed that the 5′ and 3′ carbon atoms were the site of phosphate groupings. The 3′ phosphate was esterified by the residue of the co-enzyme A molecule.

At the time of writing the structure postulated for co-enzyme A has not been confirmed by an unequivocal synthesis although a start has been made in this direction by the phosphorylation of pantetheine with phophoryl chloride. Pantetheine-2′,4′-hydrogen phosphate (cyclic) has also been prepared by the reaction of pantolactone-2-diphenyl phosphate and β-aletheine. β-Aletheine and pantolactone-2-phosphate yield the corresponding pantetheine 2′-phosphate.

Pantetheine 4′-phosphate produced by the acid hydrolysis or liver enzyme degradation of CoA has been synthesised by phosphorylation of pantetheine with dibenzyl phosphochloridate followed by removal of the benzyl groups with sodium in liquid ammonia. It was converted into co-enzyme A in the presence of adenosine triphosphate and an enzyme system from pigeon liver.

ANTIBIOTICS

Very many papers have been published during the past year dealing with all stages of the now familiar cycle of operations by which new antibiotics are discovered. This cycle, which consists of growing a micro-organism (often unidentified) upon a synthetic medium, filtering and extracting the culture filtrate, testing the extract against a range of bacteria, and finally identifying the antibiotic, has produced a bewildering variety of compounds, some highly complex, others comparatively simple.

Certain structures appear again and again; most familiar are the quinone and phthalide groupings. Extractives from the higher plants have also been tested in the same way, but results have not been encouraging; an extract from the roots of Indian carrot, for example, had a high *in vitro* activity but almost no *in vivo* activity, while the active products of *Primula malacoides* and *Buxus sempervirens* were identified as a flavone and a mixture of alkaloids, both highly toxic.

Where the antibiotic is simple in structure it is often possible to form a theory as to its mode of action. Antithiazic acid, for instance, which was extracted in 1952 from *Streptomyces virginæ*, had a high tuberculostatic activity *in vitro*. It was identified as thiazolidone-2-caproic acid. The similarity to biotin suggested that this activity was due to intervention in the synthesis of biotin by the bacterium. This was confirmed by the blocking of the *in vitro* activity by the addition of biotin, and the absence of any *in vivo* activity, there being adequate biotin within the living tissue.

The Older Antibiotics

Work has continued on the older antibiotics and related structures. Because of the known instability of β-lactam rings it has been considered that during metabolism the β-lactam ring in penicillin is cleaved and that the cleavage of this ring contributes to the antibiotic activity. Various modifications of the β-lactam ring such as its replacement by the γ-lactam or its opening between the two assymetric centres have previously been reported and the compounds shown to have no activity.

An attempt to enlarge the β-lactam ring to six members and to prepare the vinylog of penicillin has been unsuccessful although certain δ-lactams were prepared.

The most promising development in the penicillin field has been the report of the isolation from *Cephalosporium* of a substance designated as Cephalosporin N. This is believed to be a new natural form of penicillin differing from the range of penicillins already available in being active against gram negative organisms as well as gram positive, i.e. having a wider spectrum than its predecessors. The antibiotic is inactiv-

ated by penicillinase and upon acid hydrolysis gives derivatives of penicillamine. Although the new substance contains both acidic and basic groups it behaves as an acid on the ion exchange column.

Variations in the chloromycetin structure which have been tried in the past include replacement of the hydroxyl groups in the 1,3-propane diol side chain by hydrogen or alkyl, and extension of the side chain by substitution in position 2 or 3 with methyl or *p*-nitro phenyl. The dichloracetamido derivatives of DL-erythro- and threo-*p*-nitrophenylserine, and esters of these compounds with 1-*p*-nitro-phenyl-2-dichloracetamido-1, 3-propane diol, and the sulphone derivative, DL - threo - 2-dichloracetamido-1-(4-methylsulphonyl-phenyl)-1,3-propane diol (prepared from *p*-methyl-mercapto-acetophenone) have also been reported.

DDT Analogue

The discovery that the DDT analogue having certain affinities with chloromycetin—2,2,2 - trichloro-*bis*-(*p*-nitro-phenyl)-ethane—was active against murine typhus in mice has stimulated the preparation of analogues of chloromycetin in which the hydrogen atoms of the side chain at positions 1 and 3 have been replaced by methyl or *p*-nitro phenyl groups. No antibacterial or antivirus activity was exhibited.

A considerable amount of work has been concerned with phthalide and phthalic acid derivatives which frequently appear as degradation products of antibiotics. Aureomycin for instance has been shown to be readily degraded to a series of tetra-substituted benzene derivatives, the simplest of which are 3-chloro-6-hydroxy-phthalic acid and its methyl ether. Because of the assymetry of this structure two phthalides are possible, 4-chloro-7-hydroxy-3-methyl phthalide and 7-chloro-4-hydroxy-3-methyl phthalide. The ethers may be degraded to 3-methoxy-6-chloro-phthalic acid. Aureomycin itself has been dechlorinated by catalytic reduction with hydrogen in the presence of a palladium/carbon catalyst to produce a new antibiotic with similar properties to the parent material.

Although the formula of terramycin has been proved to be 4,8-diketo-1-dimethylamino - 2,5,6,10,15,17 - hexahydroxy - 15-methyl - 1,4,5,8,15,16,17,18 - octahydro - 3 - naphthacene carboxamide, work is continuing upon the interrelation of its degradation products. In 1952 the alkaline degradation products terracinoic acid and *iso*decarboxy terracinoic acid were identified as 4-carboxy-5-hydroxy-3-methyl-indanone-2-acetic acid and 7-hydroxy-3-methyl-indanone-2-acetic acid. These structures have now been confirmed by synthesis. A factory for the production of terramycin in this country has just been erected.

Streptomycin has been purified by making salts with wetting agents of the sodium alkyl sulphate type. Simple mineral acid salts can be prepared by metathesis of these wetting agent salts with mineral acids. This constitutes the only known method of crystallising streptomycin, and results suggest that it exists in at least two distinct tautomeric forms.

Mycomycin is an antibiotic, interesting because of its chemical nature rather than any bactericidal or fungicidal properties. It has been shown to be an eightfold unsaturated optically active straight chain carboxylic acid $C_{18}H_{18}O_2$. On reduction it yields tridecanoic acid. Chemical and spectral data revealed the existence of acetylenic, allenic and conjugated diene linkages suggesting the formula (-)3,5,7,8-tridecatetraene-10,12-diyne-oic acid

$(CH:C{\cdot}C:C{\cdot}CH:C:CH{\cdot}CH:CH{\cdot}CH:CH{\cdot}CH_2\,COOH)$

The 3,5 diene has a *trans-cis* stereoconfiguration.

The acid is very unstable and undergoes a unique rearrangement in dilute alkaline solution involving an allene to acetylene conversion, a dual acetylene migration and a *trans-cis* to *trans-trans* conversion yielding optically inactive isomycomycin, 3(*trans*)5-(*trans*)*n*-tridecadiene-7,9,11-triyne-oic acid

$(CH_2C:C{\cdot}C:C:C{\cdot}CH:CH{\cdot}CH:CH\,CH_2\,COOH)$

Mycomycin is the first reported example of an optically active allene of natural origin.

STREPTOMYCES ANTIBIOTICS

A new antibiotic has been isolated from the culture broth of a new species of *Streptomyces* isolated from African soil and given the name of *Streptomyces chartreusis*. It crystallises from the concentrated culture filtrate as greenish-yellow crystals and has been characterised as a weak acid having the formula $C_{15}H_{10}O_6.2H_2O$. It has been called 'chartreusin' and is active against certain gram positive organisms and mycobacteria. Its ultra-violet spectrum suggests that it is a derivative of 1,4-naphthaquinone.

An actinomycetes designated as *Streptomyces No. 11* has been shown to excrete

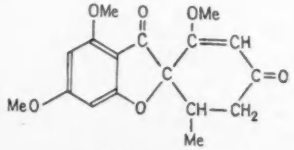
upon deep culture a substance active against both gram-positive and gram-negative bacteria. The substance has been named 'streptolin.' It was originally crystallised as the reineckate and converted to the hydrochloride. The free base has been shown to be a mixture of isomers streptolin A and B and to be represented by the formula $C_{17}H_{31-33}O_5N_5$. Streptolin A has the same elemental analysis and formula as the mixture.

Acid hydrolysis of Streptolin AB followed by chromatographic separation of the degradation products afforded five major fractions including ammonia, a basic substance having the empirical formula $C_4H_{8-10}O_2N_2$, a reducing fragment and an isomer of lysine. This last has also been isolated from the degradation products of viomycin and streptothricin. It has been called '*iso*lysine' and has the formula $C_6H_{14}O_2N_2$.

*iso*Lysine has been shown to contain a carboxyl group and a primary amino group, but no C-methyl groups; it is optically active but does not give a copper or cobalt complex. It is oxidised to succinic acid by potassium permanganate but remains unattacked by potassium periodate. There are three amino acid structures which are consistent with these facts β,ϵ-diamino caproic acid. γ,ϵ-diamino caproic acid and α-amino-methyl-δ-amino-valeric acid. The first of these has been proved to be identical with *iso*lysine by synthesis from L-ornithine using the Arndt-Eistert homologation procedure. Work on the purification of streptolins by means of ion exchange is continuing.

Antimycin A was isolated from an unidentified species of *Streptomyces* in 1949. It has the property of being able at high dilution to block an essential step in the hydrogen transport mechanism of higher aerobic organisms. Its molecular formula is $C_{28}H_{40}O_9N_2$ and it contains no strongly acidic or basic groups but a phenolic group can be detected.

Kuhn-Roth oxidation reveals the presence



dechlorogriseofulvin

of three methyl groups and two active hydrogen atoms can be detected by the use of lithium aluminium hydride.

Antimycin A hydrolyses in dilute alkaline solution at room temperature to yield antimycic acid $C_{11}H_{14}O_5N_2$ and a neutral oily fragment having the formula $C_{16}H_{28}O_4$. No other fragment could be detected to account for the missing carbon atom. Antimycic acid is atmospheric in nature, containing one weakly basic group and two acidic groups. It is a phenolic acid containing two nitro groups which are deduced to be present in the antimycin A molecule, since the ultraviolet spectra are very similar.

Acetylation yields a product with the formula $C_{15}H_{14}O_5N_2$, a *bis*-anhydro diacetate which no longer shows acidic or basic properties, and contains no phenolic group. Mild hydrolysis removed one acetyl group and replaced one molecule of water to yield the anhydro-mono-acetate, $C_{13}H_{11}O_3N_2$. The neutral fragment, a pleasant smelling oil, was found to consist of two acids, a keto-acid, $C_{11}H_{20}O_3$, and L(+)-methyl-ethyl-acetic acid. A third acid which has been obtained from the neutral fragment is believed to originate from a second antimycin component in the preparation which is known to be not entirely homogenous.

NEOMYCIN A

The activity of the culture filtrates of *Streptomyces fradiae* was at first thought to be due to a single antibiotic but fractionation of the extracts has revealed the existence of at least three separate entities known collectively as the 'neomycin complex.' In addition a further substance, active mainly against fungi, was recognised as an elaboration product and named fradicin.

Neomycin A is a minor constituent of the filtrate concentrates but the increased activity exhibited by the concentrate upon hydrolysis suggests the presence of more neomycin A in a combined form. The stability of neomycin A to acid facilitates its removal from the hydrolysate in the form of its picrate, which can be converted on isolation to the hydrochloride or sulphate. The free base can be prepared by the action of barium hydroxide on the sulphate and then acetylated or benzoylated. The acetyl and benzoyl derivatives show activity but when the former is treated with methanolic ammonia it yields an inactive N-acetyl derivative

having the formula $C_{12}H_{22}O_6N_4(Ac)_4$. Comparison of the acid degradation product of neomycin 'neamine' with neomycin A shows them to be identical.

STRUCTURE OF SOME NEW ANTIBIOTICS

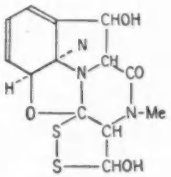
In 1951 three new antibiotics were isolated from cultures of a species of *Gliocladium* and shown to be closely interrelated. They were named aurantio and rubro-gliocladin and gliorosein. All were shown to be derivatives of *p*-benzoquinone. The first named had the formula $C_6H_6O_2(OCH_3)_2$ and the second was identified as the quinhydrone corresponding to the quinone of this formula. On reduction with sulphur dioxide both gave the same quinol which was itself isomeric with the third antibiotic. The three substances have now been identified as 2,3-dimethoxy-5,6-dimethyl-*p*-benzoquinone, the corresponding quinhydrone and a dihydro derivative of the former.

An antifungal acid, gladiolic acid, can be extracted from the culture filtrates of *Penicillium gladioli*. On alkaline rearrangement *iso*gladiolic acid is obtained. This latter has now been identified as 7-methoxy-6-methyl phthalide 4-carboxylic acid, by synthesis. Decarboxylation yields a product which has been proved by synthesis of the methyl ether to be 5-hydroxy-6-methyl phthalide. This is also given by the related compound *nor-iso*-gladiolic acid which is 5-hydroxy-6-methyl-phthalide 4-carboxylic acid. Other related compounds which have been identified are dihydro-gladiolic acid, 3-hydroxy-4-hydroxy-methyl-7-methoxy-6-methyl phthalide and cyclopolic acid (a metabolic product of *Penicillium cyclopium*) 3,5-dihydroxy-4-hydroxymethyl-7-methoxy-6-methyl phthalide.

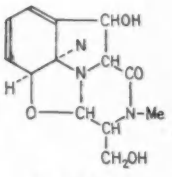
Dechlorgriseofulvin, the dechlorination product of griseofulvin, has been isolated from the culture filtrates of *Penicillium griseofulvum* and shown to be 4,6,2′-trimethoxy-6′-methyl-gris-2′-ene-3,4′-dione. The chemistry of the substances has been investigated and a sensitive test for distinguishing between the two metabolites has been devised. With strong nitric acid at room temperature dechlorogriseofulvin gives a vivid blue colour, while griseofulvin yields only a pale yellow.

ANTIBIOTICS CONTAINING SULPHUR

Thioaurin, a sulphur-containing antibiotic



gliotoxin



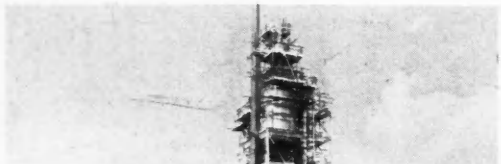
dethiogliotoxin

produced by the deep aerated culture of an unclassified strain of *Streptomyces*, is obtained as yellow needles from the ethyl acetate extract of the filtrate. It contains nitrogen and no halogen and has a molecular weight of between 290 and 400. On heating with hydrochloric acid a halogen-containing derivative is formed with the formula $C_{14}H_{12}O_2N_4S_4Cl_2$.

Oxidation of thioaurin with hydrogen peroxide gives an inactive product $C_{14}H_{12}O_3N_4S_4$. Although active against both gram-positive and negative organisms it showed little promise as an antibiotic. Another *Streptomyces* metabolite with the reported formula $C_7H_8O_2N_2S_2$ and molecular weight 214 also showed little promise and was inactivated by cysteine.

Acidification of *B. subtilis* culture filtrate has provided an antifungal substance, which has been shown to be a polypeptide containing serine. It resembles the incompletely characterised '*Aspergillus* factor.'

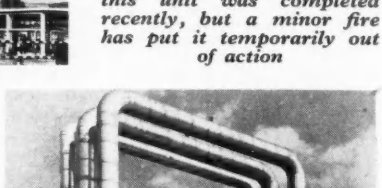
A more thoroughly investigated sulphur-containing antibiotic is gliotoxin, obtained from *Penicillium terlikowski*. The culture filtrate also contains the acetate of gliotoxin although acetylation of the latter has not proved possible. Gliotoxin may be desulphurised by treatment with amalgamated aluminium to dethiogliotoxin $C_{13}H_{16}O_2N_2$. On treatment with hot hydrochloric acid this is converted to anhydro-dethiogliotoxin $C_{13}H_{14}O_2N_2$. A product $C_{13}H_{14}O_2N_2$, given on heating the latter with acetic anhydride, has been identified as 2,3-dimethylpyrazinoindole 1.4 dione, identical with that produced by the direct reduction of gliotoxin with phosphorus and hydriodic acid. Reduction with zinc and hydrochloric acid gives $C_{13}H_{16}O_2N_2$ and catalytic reduction over Raney nickel produces a compound $C_{13}H_{20}O_4N_2$ corresponding to the tetrahydro derivative of dethiogliotoxin.



CORYTON—

A view of the Thermofor continuous percolation unit, the first of its kind in the world, at the Coryton refinery of the Vacuum Oil Co. Ltd. Construction of this unit was completed recently, but a minor fire has put it temporarily out of action

ONE OF BRITAIN'S NEW OIL REFINERIES



Expansion bends in the main steam lines frame the furfural unit. This unit removes impurities from lubricating oil stocks and ensures a high viscosity index in the finished products. It came into operation at the beginning of August, 1953

The crude distillation unit viewed from the north. This unit processes 20,000 barrels of crude oil per day; it divides the crude into a range of fractions, and also divides the heavy fractions which are used for the production of lubricating oil

Progress in Chromatography

by G. S. EGERTON, M.Sc., Ph.D., F.T.I.

(Department of Textile Chemistry, Manchester College of Technology)

THE early origins of the history of chromatography appear at the present time to be shrouded in a certain amount of obscurity. Thus, the classical pioneer experiments of Twsett (1906), who has been generally regarded as the inventor of adsorption chromatography, are now considered as being preceded by the somewhat similar work carried out at the end of the 19th century, first by the American chemist, Day, and later by two German chemists, Engler and Albrecht. Nevertheless, this method of adsorption analysis seems to have been almost completely neglected for many years, until in 1931 it was used by Kuhn, Winterstein and Lederer for the separation of a mixture of carotenoids. Since this time progress in chromatography has been made with the utmost rapidity and interest in this now very valuable chemical technique has been increasingly maintained. It is probably true to say that this new technique has advanced progress in every branch of chemistry and that its possibilities have still not been completely exhausted. One of its most attractive virtues lies in the fact that unlike many other modern techniques, it does not involve the use of highly complicated and expensive equipment.

In a recent paper Tiselius (*Endeavour*, 1952, **11**, 5) has discussed the principles underlying the use of chromatographic methods, and has described the ways in which chromatograms may be developed. He has also compared the different techniques involved in the practice of partition and paper chromatography, column chromatography, and of ion exchange chromatography. The uses of the ion exchange method were extensively discussed in this journal just over two years ago (THE CHEMICAL AGE, 1951, **64**, 743), and owing to limitations of space it is therefore not proposed to devote as much attention as might otherwise have been desired to this branch of chromatography in the present report.

A most useful review of the innovations in the principles and the practice of partition, paper and column chromatography covering the period from 1950 to 1952 has been published by Moore and Stein (*Ann. Rev. Biochem.*, 1952, **21**, 521). This paper also includes over 200 references to the literature. The principles of chromatography and the many applications of paper chromatography have also been extensively reviewed by Ylstra (*Chem. Weekblad.*, 1952, **48**, 793, 813; 1953, **49**, 316).

An account of the use of chromatography in legal medicine has been compiled by Pérez Argilés and Bastero Beguiristain (*Arch. Med. Exptl.*, 1952, **15**, 129) in which the methods and various apparatus of chromatography are described. These workers have related how the pigments contained in human hair were separated chromatographically after the hair had been treated with caustic potash. The pigment layers were examined in natural light and in Wood's light. It was found possible to separate the alkaloids contained in visceral extracts chromatographically, but the use of ultra-chromatography was not found to be practical. However, blood pigments and the degradation products of haemoglobin were successfully examined by this latter method. The use of semi-automatic instruments in paper chromatography has been described by Müller (*Analyst*, 1952, **77**, 933).

RF VALUES

The introduction of paper chromatography by Consden, Gordon and Martin in 1943 may be considered to be a natural development of the partition column of Martin and Synge. The mechanism of the latter column is thought to consist mainly of partition between two phases, and this concept transferred to chromatography on filter paper suggests a partition between a mobile solvent phase and a stationary phase composed of a cellulose-water complex. Paper chromatography can be described in brief as a process in which a small quantity of material—usually a mixture—is placed on a limited area of a piece of filter paper, which is then irrigated by a solvent and to which after the elapse of some time methods for the detection of the material are applied.

Under suitable experimental conditions the material will be found to have migrated from its point of application and, if it is a mixture, to have separated partially or wholly into its components.

The term R_F value was introduced in order to specify the particular position taken up by a substance on a paper chromatogram. It represents the ratio of the distance travelled by the substance to that of the solvent front, measured from the point of application of the material. Kowkabany and Cassidy (*Anal. Chem.*, 1952, **24**, 643) have made a study of the various factors that may influence R_F values in ascending paper chromatography using amino acids as solutes. Two simple R_F scales, triangular and spiral, have been developed by Nettleton and Mefferd (*Anal. Chem.*, 1952, **24**, 1687) which enable direct readings to be made of R_F values and which eliminate cumbersome measurements and computations.

R_F values in paper chromatography have been studied by Zimmermann (*Z. anal. Chem.*, 1953, **138**, 321), who considers that these values show the accuracy and dependence on various factors of the chromatographic tests. The values depend upon (1) the development phase, (2) the kind of paper, (3) the direction of the paper fibres, (4) the manner of development, (5) the wave length used for the development, (6) the distance of the starting line from the reservoir used for the development phase, (7) the concentration of the solution, (8) the other substances present, and (9) the temperature. These points are discussed by the author in some detail.

An important study of the mechanism of paper chromatography has been made by Horner, Emrich and Kirschner (*Z. Elektrochem.*, 1952, **56**, 987). These workers examined the interaction of cellulose with solvents by determining refractometrically the composition of a solvent after percolation through a paper strip. It was found that water was preferentially taken up from aqueous solutions of butyl alcohol, phenol, β-picoline and *iso*-propyl alcohol, and that oven-dried papers imbibed more water than air-dried paper. The addition of amino acids or potassium iodide increased the binding of the water. The results were held to show that a cellulose-water complex, or more likely a cellulose-water organic solvent complex, is formed, probably predominantly in the amorphous regions of the cellulose. When an equilibrium mixture of water and phenol was allowed to rise in a paper strip, the phenol content, determined iodimetrically after cutting the paper into segments, increased towards the top. The R_F values of amino acids increased with a rise in the water content of the solvents, and the resolution decreased. The R_F values decreased if the paper was first equilibrated with the solvent system. Pretreatment of the paper with diazomethane, designed to methylate the carboxyl groups if any were present in the cellulose, had no effect on the R_F values. It was concluded from these results that ion exchange and adsorption play no significant part in paper chromatography. The mechanism is considered to be dependent upon a partition between the cellulose-water-organic solvent complex and the mobile phase.

GRADIENT ELUTION ANALYSIS

A new and widely applicable method for the chromatographic analysis of mixtures of compounds has been outlined by Alm, Williams and Tiselius (*Acta Chem. Scand.*, 1952, **6**, 826). Described as gradient elution analysis it consists in the use of an eluting medium whose composition is continuously changed by the addition of a solvent of high eluting power at a known rate to a known initial volume of another solvent of lower eluting power in a mixing chamber which drains into the column. The theoretical side of this method is discussed by the authors, and the effects of such factors as the spreading of zones, the separation of components, and the pretreatment of the adsorbent are considered. In a subsequent paper (Alm, *Acta Chem. Scand.*, 1952, **6**, 1186) this method of gradient elution analysis was used in an analysis of a dextrin sample containing dextrin and a mixture of mono to hexa saccharides. Experiments on the resolution of amino acids and of peptides in cases where the standard procedures were unsatisfactory have been described by Williams (*Analyst*, 1952, **77**, 905), who has suggested that gradient elution analysis overcomes some of these difficulties, although it may present others. In the gradient elution method described by this author, there is also a temperature gradient along the column so that the experimental procedure resembles a continuous fractional crystallisation process.

An automatic device has been described by Donaldson, Tulane and Marshall (*Anal. Chem.*, 1952, **24**, 185) that permits the delivery of solvents that gradually increase in polarity during the course of a chromatographic separation. This device is useful for the better resolution of organic acids on columns of silica gel. In a subsequent paper (Marshall, Donaldson and Friedberg, *Anal. Chem.*, 1952, **24**, 773) the authors refer to the fact that the literature lacks any approach for calculating chromatographic positions (R_F values) for such a method involving progressively changing solvents. A technique is described for computing these chromatographic positions, and the migration of several organic acids in several solvent systems is analysed by the simple equation derived. It is claimed that this technique allows the chromatographic procedure to be scaled upward or downward, and the possibilities of this newer type of chromatography are illustrated by experimental data.

Drake (*Arkiv. Kemi*, 1952, **4**, 401) has described an apparatus in which chromatography is combined with the automatic recording of electrolytic conductivity, and has shown how it may be used in the analysis of amino acids and peptides. A continuous automatic recording of the resistances of eluates from adsorption columns is described. The apparatus was successfully used in a frontal analysis of a mixture of glycine and arginine, and in a carrier displacement analysis of a mixture of methionine, leucyldiglycine and phenylalanine.

Many of the difficulties involved in the chromatographic separation of members of a polymeric series are the result of the inability of the larger molecules to enter the porous materials by chromatography. Electrokinetic ultrafiltration is recommended by Mould and Synge (*Analyst*, 1952, **77**, 964) as a means of overcoming this difficulty. A preliminary account is given of experiments in which collodion membranes were used and separations were obtained of a series of enzyme-synthesised, straight chain polysaccharides. After a substance has been separated by chromatography or electrophoresis the quantity present can be determined by measuring the amount of light absorbed by the substance (Campbell and Simpson, *Chemistry & Industry*, 1953, 342).

It is not necessary in the method described to separate or remove the substance from the filter paper, but it is essential that the substance should be either coloured or capable of quantitative conversion to a coloured substance by a reaction that need not be specific. A Unicam SP500 quartz spectrophotometer is used in these measurements. The coloured spot is tracked point by point so that a plot can be made of apparent optical density against distance along the strip. The area under the peak above the background absorption due to the filter paper itself has been found to be proportional to the total amount of coloured material present to within about 5 per cent. As small an amount as 10^{-6} gm. of coloured compound can be detected in this manner. An alternative method is to measure the absorption in the infra-red part of the spectrum. Hoyer (*Kolloid-Z.*, 1952, **127**, 166) has described a special flow cell for use with the Perkin Elmer double beam infra-red spectrometer. With this cell, liquid from a chromatographic tube can be analysed in various ways, either directly, intermittently or continuously.

Some attention has been devoted to the use in chromatography of paper containing acidic groups. Wieland and Berg (*Angew. Chem.*, 1952, **64**, 418) found that paper with a carboxyl content of between 1 per cent and 4 per cent gave promising results in the separation of amino acids, amines and inorganic ions. Lautsch, Manecke and Broser (*Z. Naturforsch.*, 1953, **8b**, 232) also showed that charged groups, such as carboxyl, sulphonic acid, etc., could be introduced into filter paper strips and these strips used for the separation of amino acids on paper either chromatographically or electrophoretically. It was found that sometimes the results were better than those obtained using ordinary filter paper.

GAS-LIQUID PARTITION CHROMATOGRAPHY

James and Martin (*Analyst*, 1952, **77**, 915) have described a convenient apparatus in which a gas-liquid partition column is used for the separation of volatile acids and bases. Using a four foot column and a stationary phase consisting of a mixture of a liquid silicone and stearic acid it was found possible to separate all normal and *iso* acids from formic acid to undecanoic acid. With a longer column (11 ft.) all the isomers of valeric acid could be resolved. In the

separation of volatile bases, advantage was taken of their hydrogen bonding power so that by using stationary phases consisting of hydrogen bonding and non-hydrogen bonding substances, it was possible to separate a number of amines. This method of gas-liquid partition chromatography has been applied in the separation and micro-estimation of a mixture of ammonia and the three methyl-amines, whose microanalysis by other means is somewhat difficult (James, Martin and Howard Smith, *Biochem. J.*, 1952, **52**, 238). James (*Biochem. J.*, 1952, **52**, 242) has also described the use of gas-liquid chromatography in the separation of volatile aliphatic amines and of the homologues of pyridine. Various methods for the separation and micro-determination of primary, secondary and tertiary aliphatic amines are discussed. Columns having polar and non-polar liquid phases can be used to identify an unknown amine as a primary, secondary or tertiary compound. A large number of amines containing from 1 to 12 carbon atoms were separated on four foot columns, with the exception of *iso* and secondary butylamine, and di-*iso* and di-*sec.* butylamine. Details are given of the separation of pyridine, α-picoline, *p*-picoline, 2, 6-lutidine, 2, 4-lutidine, and 2, 4, 6-collidine at 137° using liquid paraffin as the stationary phase, but it was not found possible to separate β-picoline and *p*-picoline by this means.

GAS CHROMATOGRAPHY

Examples are given by Griffiths, James and Phillips (*Analyst*, 1952, **77**, 897) of the chromatographic separation and analysis of volatile organic mixtures from charcoal columns at 100° with results that are accurate to within 1 per cent. An account is also given of some elution analyses involving the use of partition columns. The separations have been followed by a thermal conductivity cell, but a very promising device depends on surface potential changes and is sensitive to extremely low concentrations of vapour. Its use enables gas chromatographic methods to be extended to relatively non-volatile materials.

James and Phillips (*J. Chem. Soc.*, 1953, 1600) have described an apparatus for the analysis and quantitative chromatographic separation of volatile substances by adsorption and partition techniques. The nearly ideal behaviour of gases results in greater efficiency than in liquid chromatography. Gases are used as the moving phase and heated charcoal is used in the adsorption column. Silicone fluid, nitrobenzene, tricresyl phosphate, or dibutyl phthalate supported by Celite is used in the partition columns. A stream of nitrogen is bubbled through the analysis mixture and the saturated nitrogen passed through the chromatographic columns. The adsorbed vapours are displaced, and in general the separation follows the order of the boiling points, so that a given vapour displaces the vapours of other compounds with a lower boiling point. The displaced vapour is measured in a thermal conductivity recorder which plots a series of peaks as the various components are displaced. It is possible to detect 0.02 gm. of a volatile substance with an accuracy of 1 per cent.

SPRAYS

Müting (*Naturwissenschaften*, 1952, **39**, 303) has found that with about 25 different amino acids characteristic colours are obtained on filter paper by spraying with a dilute solution of the sodium salt of 1, 2-naphthoquinone 4-sulphonic acid in aqueous sodium carbonate and drying at room temperature. The same colours are also obtained by spraying the paper chromatograms of protein hydrolysates. The colour differentiation is claimed to be better than when using ninhydrin, especially for proline and hydroxy-proline, and the sensitivity is stated to be almost equal to that obtained with ninhydrin. Giri and Nagabhushanam (*Naturwissenschaften*, 1952, **39**, 548) also give the typical colours produced by 28 amino acids and seven peptides with this naphthoquinone reagent, and consider that the advantages of this reagent over ninhydrin are in definition of bands and distinction of colour. Proline and hydroxy-proline give orange colours which can be eluted readily with a small quantity of water and used for determination in a colorimeter.

In the paper chromatographic analysis of imidazoles, Ames and Mitchell (*J. Amer. Chem. Soc.*, 1952, **74**, 252) recommend that the air-dried chromatograms be sprayed lightly with diazosulphanilic acid reagent, and the colour developed with a light spray of dilute sodium carbonate solution. Mukherjee and Srivastava (*Nature*, 1952, **169**, 330) have detected sugars on paper chromatograms by spraying with the filtrate from a mixture of *p*-anisidine and phos-

phoric acid in alcohol. Sattler and Zerban (*Anal. Chem.*, 1952, **24**, 1862) found that common hexoses and pentoses gave orange spots on a light background when the chromatogram was sprayed with an alcoholic solution of *p*-aminohippuric acid and heated. Under ultra-violet radiation these spots fluoresced strongly. A spray reagent containing phthalic acid was used to show the presence of glucose and fructose and could be used to detect the presence of other reducing and easily hydrolysed non-reducing sugars. Hockenhull (*Nature*, 1953, **171**, 982) observed that in the chromatographic separation of sugars upon filter paper impregnated with borax buffer, carbohydrates showed up prominently on spraying with aqueous ethyl alcohol containing phenolphthalein or phenol red made just alkaline with caustic soda. A spraying agent of this type was developed which gave good definition with a number of sugars.

ORGANIC ANALYSIS

Until the advent of paper chromatography the problem of the analysis of amino acids, particularly of the complex mixtures resulting from the hydrolysis of proteins, presented great difficulty. It was largely the result of the comparative ease with which complex mixtures of amino acids could be separated by paper chromatography that this method was successfully used by many workers in the analysis of other complex mixtures.

(a) Amino Acids

Sjöquist (*Acta Chem. Scand.*, 1953, **7**, 447) has identified a number of naturally occurring amino acids as the phenyl thiohydantoins by paper chromatography. A method for the identification of thiohydantoins (prepared from a number of amino acids) by paper chromatography has also been described by Edward and Nielsen (*Chemistry & Industry*, 1953, 197). Levy and Chung (*Anal. Chem.*, 1953, **25**, 396), have described an improved method for the two dimensional chromatographic analysis of amino acids on buffered papers. Details of a method using ninhydrin for the determination of the amount of amino acids present on paper chromatograms have been published (Bode, Hübener, Brückner and Hoeres, *Naturwissenschaften*, 1952, **39**, 524). The spots on equal size paper are cut out as well as blank paper standards, the paper is eluted with methyl alcohol, and optical densities determined with a quartz spectrophotometer. Good results were obtained with a number of amino acids, but it is reported that proline was difficult to determine by this method. A solvent prepared by refluxing a mixture of formic acid, butyl alcohol and water, then adding a further quantity of water, and using the upper solvent layer upon separation after 24 hours, was found to give a good separation in the chromatographic analysis of amino acids in sugar cane (Wiggins and Williams, *Nature*, 1952, **170**, 279). This solvent was also used successfully in the separation of a mixture of glucose, fructose, sucrose and a number of other sugars.

(b) Sugars

Yoda (*J. Chem. Soc., Japan, Pure Chem. Sect.*, 1952, **73**, 18) has described the use of paper chromatographic methods in the detection of non-reducing sugars and polyhydric alcohols. The separation of sugar mixtures by combined electrophoresis and paper chromatography has been studied by Micheel and van de Kamp (*Angew. Chem.*, 1952, **64**, 607). The mixtures were used as complexes with boric acid. Electrophoresis was carried out during the downflow of the solution on filter paper. Fractions were collected and then chromatographed separately. Paper partition chromatography was also used by Edward and Waldron (*J. Chem. Soc.*, 1952, 3631) in the detection of deoxy sugars, glycals and methyl pentoses. Bayly and Bourne (*Nature*, 1953, **171**, 385) have reported that the R_F values of oligo-saccharides can be increased (eightfold for disaccharides) by converting them into N-benzylglycosylamine derivatives. The chromatographic analysis of mixtures of methyl fructosides, prepared by reacting fructose with a mixture of hydrochloric acid and methyl alcohol, has been described by Augestad, Berner and Weigner (*Chemistry & Industry*, 1953, 376).

(c) Organic Acids

Denison and Phares (*Anal. Chem.*, 1952, **24**, 1628) have described a rapid chromatographic method for the separation of tartaric, oxalic, citric, lactic and a number of other organic acids. The method requires less than two hours for the satisfactory development of the paper chromatogram. A micro method involving two dimensional chromatography has been used by Cheftel, Munier and Macheboeuf (*Bull. Soc. Chim.*

Biol., 1951, **33**, 840; 1952, **34**, 380) for the separation and identification of a number of non-volatile, water-soluble aliphatic acids, including oxalic, malonic, succinic, adipic, citric and lactic acids. A number of organic acids have also been identified by paper chromatography by Jones, Dowling and Skraba (*Anal. Chem.*, 1953, **25**, 394). Tartaric, citric, malic and malonic acids were separated by Airan, Joshi, Barnabas and Master (*Anal. Chem.*, 1953, **25**, 659) using circular paper chromatography. Vandenheuvel and Hayes (*Anal. Chem.*, 1952, **24**, 960) have published details for the separation on a column of silicic acid of monocarboxylic acids containing from two to twelve carbon atoms and dicarboxylic acids containing from four to ten carbon atoms. The purity of chloroacetic acid has been checked by a chromatographic method (Smith, Mueller and Rogers, *Anal. Chem.*, 1952, **24**, 1117). Unsaturated organic acids, especially linoleic and linolenic acids, have been separated chromatographically by Modica and Rossi (*Chim. anal.*, 1952, **34**, 271). Paper chromatography has also been used in the separation and analysis of barbituric acids (Grieg, *Nature*, 1952, **170**, 845), nicotinic acids (Allouf and Munier, *Bull. Soc. Chim. Biol.*, 1952, **34**, 196), and nucleic acid components (Edström, *Nature*, 1951, **168**, 876).

(d) Miscellaneous Organic Compounds

Paper chromatography has been used in the separation and identification of phenols and phenolic acids (Durant, *Nature*, 1952, **169**, 1062), polyphenols (Roberts and Wood, *Biochem. J.*, 1953, **53**, 332), and methylol phenols (Freeman, *Anal. Chem.*, 1952, **24**, 955, 2001). According to a method described by Jerchel and Jacobs (*Angew. Chem.*, 1953, **65**, 342) a mixture of alkylpyridines can be analysed by first oxidation with selenium dioxide, and then separation of the resulting pyridine carboxylic acids by paper chromatography. The acids are rendered visible in ultra-violet radiation by spraying the paper chromatogram with a dilute solution of fluorescein. Details are given of the analysis of a commercial mixture of pyridine bases.

A brief review of the results obtained in the separation of carotenoids and chlorophylls by paper chromatography in one and two dimensions has been provided by Bauer (*Naturwissenschaften*, 1952, **39**, 88). Other uses to which paper chromatography has been put include the separation and detection of acetone and methyl ethyl ketone (Mentzer, Molho and Molho-Lacroix, *Bull. Soc. Chim.*, France, 1953, 636), α-keto acids (Hockenhull, Hunter and Herbert, *Chemistry & Industry*, 1953, 127), thioureas (Kjaer and Rubinstein, *Acta Chem. Scand.*, 1953, **7**, 528; *Nature*, 1953, **171**, 840), sulphonamides (Steel, *Nature*, 1951, **168**, 877), methyl esters of stearic, oleic, linoleic and linolenic acids (Kurtz., *J. Amer. Chem. Soc.*, 1952, **74**, 1902), flavone derivatives (Paris, *Bull. Soc. Chim. Biol.*, 1952, **34**, 767; Casteel and Wender, *Anal. Chem.*, 1953, **25**, 508), the dinitrobenzoates of volatile aliphatic alcohols (Meigh, *Nature*, 1952, **169**, 706), guanidine compounds (Makisumi, *J. Chem. Soc., Japan, Pure Chem. Sect.*, 1952, **73**, 737), and purines, pyrimidines and nucleosides (Löfgren, *Acta Chem. Scand.*, 1952, **6**, 1030).

INORGANIC ANALYSIS

A useful account by Burstall and Kember (*Ind. Chemist*, 1950, **26**, 400) described the technique involved in the chromatographic separation of inorganic compounds using paper strips or columns of cellulose pulp with organic solvents as eluents. This paper also includes a list of the ions that can be separated by these methods. Another valuable review of the analysis of inorganic compounds by paper chromatography has been provided by Pollard and McOmie (*Endeavour*, 1951, **10**, 213). More recently some experimental details have been given by Surak *et al.* (*J. Chem. Educ.*, 1952, **29**, 144; 1953, **30**, 20) which can be used for the separation by paper chromatography of inorganic cations belonging to Groups I to V. The qualitative and quantitative analysis of inorganic ions has also been investigated by Lederer (*Anal. Chim. Acta.*, 1951, **5**, 185, 191; 1952, **7**, 458), and Venturello and Ghe (*Anal. Chim. Acta*, 1952, **7**, 261).

Fernando and Phillips (*Anal. Chem.*, 1953, **25**, 819) have used paper impregnated with various quinolinols, with pyridine or ethyl alcohol as eluent, in the separation of bivalent ions of bismuth, cadmium, cobalt, copper, nickel and zinc, and the trivalent ions of iron and aluminium. Using paper impregnated with 4-methyl-8-quinolinol it was possible to obtain complete migration of all these ions with pyridine.

Details have been given of methods suitable for the chromatographic separation of

copper, cobalt and nickel (Airan and Barnabas, *Science and Culture*, 1953, **18**, 438), and for the determination of copper in zinc, and zinc in cadmium (Oka and Murata, *J. Chem. Soc., Japan, Pure Chem. Sect.*, 1951, **73**, 494, 496). Pollard, McOmie, Stevens and Maddock (*J. Chem. Soc.*, 1953, 1338) have described a method for the analysis of molybdenum as found in samples of steel, and for its separation from cobalt, chromium, copper, iron, nickel, manganese and vanadium. Details are also given of the analysis of copper and iron as found in brass.

The separation by paper chromatography of molybdenum, vanadium and chromium (Lacourt *et al.*, *Michrochemie ver. Mikrochim. Acta*, 1953, **40**, 268; *Compt. rend.*, 1952, **234**, 2365), and of uranium, vanadium and chromium (Harasawa and Sakamoto, *J. Chem. Soc., Japan, Pure Chem. Sect.*, 1953, **74**, 285) has also been described. In a series of papers, Williams *et al.* (*J. Chem. Soc.*, 1952, 1497, 3155, 3399; *Analyst*, 1952, **77**, 983) have studied the separation of tantalum and niobium by chromatography on cellulose, and have established methods for the quantitative determination of these metals. The same author (*Analyst*, 1952, **77**, 293, 297) has also studied the use of columns of alumina and cellulose for the determination of uranium and thorium in minerals and ores.

Other chromatographic investigations include the separation and determination of iron, aluminium and titanium (Lacourt, Sommereyns and Wantier, *Analyst*, 1952, **77**, 943), of zirconium and hafnium (Kember and Wells, *Chemistry & Industry*, 1952, 1129), the noble metals (Shibata and Uemura, *J. Chem. Soc., Japan, Pure Chem. Sect.*, 1951, **72**, 757), thallium (Diller and Rex, *Z. Anal. Chem.*, 1952, **137**, 241), arsenic (Harasawa and Sakamoto, *J. Chem. Soc., Japan, Pure Chem. Sect.*, 1952, **73**, 611), potassium, sodium, magnesium and lithium (Harasawa and Sakamoto, *J. Chem. Soc., Japan, Pure Chem. Sect.*, 1952, **73**, 614), and lanthanons (Pollard, McOmie and Stevens, *J. Chem. Soc.*, 1952, 4730).

It has also been possible to use paper chromatography for the identification of ortho, meta and pyro phosphates (Ando, Ito, Ishii and Soda, *Bull. Chem. Soc., Japan*, 1952, **25**, 78). The presence of arsenates and nitrates does not interfere with the identification. The analysis of anions by paper chromatography has also been studied by Nakano (*J. Chem. Soc., Japan, Pure Chem. Sect.*, 1953, **74**, 56).

RECENT BOOKS

During the last year or so a number of useful books dealing with various aspects of chromatography have been published, and since some of them may be of interest to the reader details are given below:—

'Adsorption and Chromatography,' by H. G. Cassidy. Interscience Publishers Inc., 1951. Pp. 360. Price 56s.

'A Guide to Filter Paper and Cellulose Powder Chromatography,' by J. N. Balston and B. E. Talbot. H. Reeve Angel & Co. Ltd. and W. & R. Balston Ltd., 1952. Pp. 145. Price 8s.

'Paper Chromatography,' by R. J. Block, R. Le Strange and G. Zweig. Academic Press Inc., 1952. Pp. 195. Price 36s.

'Chromatographic Methods of Inorganic Analysis,' by F. H. Pollard and J. F. W. McOmie. Butterworths Scientific Publications, 1953. Pp. 192. Price 30s.

'Practical Chromatography,' by R. C. Brimley and F. C. Barrett. Chapman and Hall Ltd., 1953. Pp. 128. Price 15s.

'Chromatography: A Review of Principles and Applications,' by E. and M. Lederer. Elsevier Publishing Co., 1953. Pp. 460. Price 60s.

Interests Acquired

The management and some other shareholders of Northern Wood Preservers Ltd. have purchased the substantial stock interests in the company held by the Great Lakes Paper Co., R. J. Prettie, president and general manager, announces. Great Lakes Paper originally acquired its holdings in Northern Wood early in 1950 in an effort to accomplish maximum utilisation of timber resources. G. E. Kernohan, vice-president, and S. F. Mack, general superintendent, have been elected to the board of Northern Wood, replacing the Great Lakes Paper representatives. Mr. Prettie reports full operation at the Northern Wood Preservers plant and capacity business at the tar and chemical subsidiary during the past year. He said tar and chemical business is likely to continue active this year.

Chemical Stoneware Tests

New British Standard

FIFTEEN years after the first publication of BS. 784 'Testing Chemical Stoneware,' a revision has been published by the British Standards Institution. During that period certain of the tests specified have been found to be of academic interest only and others have been superseded by newer tests.

The new document gives the following test methods with full details of apparatus, test piece and procedure:—

1. *Determination of resistance to compression.*—The size of test piece has been more closely defined because results seem to vary with differing sizes and shapes.

2. *Determination of transverse strength.*—This test is basically the same as the method for determination of resistance to cross bending. It is felt, however, that it does not give a true modulus of rupture for stoneware, and although a reliable comparison of stoneware can be obtained with it, the results should not be used for design calculations on stoneware apparatus without considerable factors of safety.

3. *Determination of resistance to abrasion.*—A more accurate method has been adopted.

4. *Determination of true specific gravity.*—Only minor changes have been made, but specific gravity has been substituted for density.

5. *Determination of apparent specific gravity, apparent porosity and water absorption.*—The use of paraffin for this test has been replaced by water. Instead of necessitating a separate test, the water absorption is calculated from the result.

6. *Determination of thermal expansion.*—Owing to errors caused by the casual heating of the dial gauge and support on the older vertical apparatus, this test has been superseded by one using a horizontal test piece.

7. *Determination of resistance to thermal disruption.*—This is a new test based on the lowering of the transverse strength of stoneware after subjecting it to specified degrees of thermal shock.

8. *Determination of acid soluble iron.*—A more accurate method has been adopted.

9. *Determination of resistance to acid.*—The resistance of stoneware to acid attack is extremely high. It is therefore only possible to obtain a result by breaking down the sample to get an extremely large surface area and by using concentrated acid. This test must not be confused with 'Acid solubility' as given in some other British Standards obtained by using a single piece immersed in weak acid.

Copies of this standard may be obtained from the British Standards Institution, Sales Branch, British Standards House, 2 Park Street, London, W.1, price 3s.

Potash Exports Resumed

First Shipment Leaves Israel

THE first shipment of potash exported since the establishment of the State of Israel left Israel recently. Part of a 3,000-ton British order, this consignment weighed 250 tons. The resumption of potash export will provide many thousands of additional dollars to balance Israel's international trade. Potash had been a major export before the Arab-Israel war.

This shipment represents the culmination of efforts to rehabilitate the Dead Sea industry which was disrupted in 1948. Until the end of 1947, Palestine Potash Ltd. carried on operations at both the northern and southern ends of the Dead Sea. The northern plant was lost during the hostilities. The southern plant suspended operations and remained idle until the middle of 1952. At that time a new company, 'Mifaley yam Ha-Melech Ltd.' (Dead Sea Works) was incorporated.

This company acquired the assets of Palestine Potash Ltd. and resumed the exploitation of the natural deposits of the Dead Sea. The completion, last spring, of a 48-mile highway from Beersheba to Sodom, constructed by the Government of Israel, was an essential step in the re-activation of these works. This road provides easy transportation to and from the coast.

Before the second world war, Palestine Potash produced 100,000 tons annually—one twelfth of the world production. Of this amount, 60,000 tons was the potential of the southern plant. Following the rehabilitation of the plant works this year, it is expected that production will reach 200 tons per day. A net production of 100,000 tons has been planned for 1954. The target for 1958 is 300,000 tons.

Sulphuric Acid Output to Capacity

Rising Trend of German Chemical Sales

THERE has been a slowing down in the rising trend of German chemical sales both at home and abroad; the official production index for chemicals in November was 6 per cent higher than in the corresponding month of 1952, and the percentage share of the chemical industry in German industrial turnover and export sales has been declining slowly as other industries forged ahead more swiftly. The outlook however is viewed with confidence by German chemical manufacturers, as remarks at recent shareholders' meetings indicate, and a further increase in production is forecast for the new year.

Sulphuric acid makers may find it difficult to provide greatly increased supplies if demand rises substantially. After rising from 329,000 metric tons (as SO_3) in July-September, 1952, to 378,500 tons in the corresponding period of 1953, the German sulphuric acid capacity is now fully utilised, and a further increase awaits the completion of plant extensions now under way. German sulphuric acid production, which almost doubled between 1946 and 1948 and again between 1948 and 1951, has been rising at a slower rate since. Between the third quarters of 1952 and 1953 the increase amounted to 15 per cent, compared with a rise by 21.5 per cent in German chemical production as a whole.

Dependence on Imports

West German sulphuric acid makers depend chiefly on imported pyrites, mostly from Spain and other Mediterranean sources, for their raw material and did not therefore suffer greatly from the world sulphur shortage. Unlike their colleagues in Eastern Germany, they did not experiment with methods of utilising indigenous materials, but concentrated their efforts on improving the pyrites roasting processes, apparently with some success. Maximum use is made of opportunities for obtaining acid in the metal smelting industry while smaller amounts are recovered from spent oxide. No less than three-tenths of the whole German acid output is used for making ammonium sulphate while superphosphates account for another one-eighth of consumption.

The use of benzole for technical and industrial purposes in Germany—now at a rate of about 200,000 tons a year—is certain to increase as larger quantities are required for styrene, phenol, plastics, fibres, insecticides and detergents, and steps are being taken to provide additional plant for refining and distilling. The Scholven-Chemie AG is extending its catalytic pressure refining plant, and Harpener Bergbau AG is erecting a new plant employing the Scholven process. The combined capacity of the two companies will be 300,000 tons a year, sufficient to deal with the bulk of the benzole output in the Ruhr area. Most of the refined benzol from the Scholven plant—between 12,000 and 15,000 tons a month—will be passed on to a new continuous distillation unit now being erected by Redestillationsgemeinschaft GmbH on the outskirts of Gelsenkirchen.

First in the World

An underground pipeline system, described as the first benzol pipeline in the world, is being built now to connect the plant of Scholven-Chemie AG with the harbour of Gelsenkirchen where the new distillation plant is situated and to link the latter with the plant of Chemische Werke Hüls 13 miles away. Storage tanks to absorb seasonal surpluses are also provided at Gelsenkirchen. During the second phase of the construction work plant for the production of pure toluol, xylol and arsols will be erected. The arsols are expected to meet the demand for a high-grade aromatic solvent with a high boiling point.

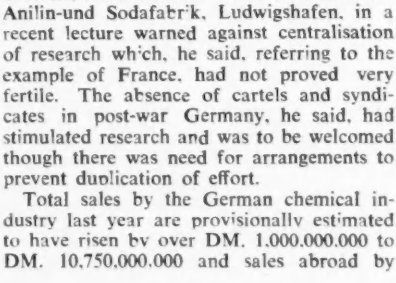
The production of synthetic rubber by Chemische Werke Hüls at Marl still appears to meet with difficulties. At the recent general meeting of the company it was stated that the production of butadiene is uneconomical owing to increased coal and electricity charges. Buna production, which in 1952 was at a monthly rate of 350 tons, is apparently still below the ceiling of 500 tons a month fixed two years ago when the Allied authorities consented to the resumption of synthetic rubber production. In the light of recent experience the German experts endorse French and Italian estimates that the smallest economical unit for the produc-

tion of synthetic rubber is one with an output capacity of 25,000-30,000 tons a year.

At present Buna is one of the least important products of Chemische Werke Hüls. Research is concentrated on other subjects, in particular the improvement of the thermal production process for acetylene and ethylene from hydrocarbons and on detergents, textile agents and basic materials for fully synthetic fibres. About DM. 90,000,000 have been spent at Marl on extending production between 1945 and 1952, and in 1953 another DM. 30,000,000 were invested in new plant while sales increased by about 20 per cent.

While the majority view in the German chemical industry still favours maximum co-operation and centralisation in research, this opinion is not apparently shared by all industrial leaders. Professor Dr. h. c. Carl Wurster, chairman of the board of Badische Anilin-und Sodafabrik, Ludwigshafen, in a recent lecture warned against centralisation of research which, he said, referring to the example of France, had not proved very fertile. The absence of cartels and syndicates in post-war Germany, he said, had stimulated research and was to be welcomed though there was need for arrangements to prevent duplication of effort.

Total sales by the German chemical industry last year are provisionally estimated to have risen by over DM. 1,000,000,000 to DM. 10,750,000,000 and sales abroad by DM. 400,000,000 to DM. 2,200,000,000. The quantitative increase in production was even greater—17 per cent, compared with 11 per cent increase in value. Employment in the German chemical industry rose steadily this year, but by September the industry's manpower was still only three per cent above 1952. Many firms report that new products launched since 1948 account for 20-30 per cent of their total output. Big new schemes of expansion have been prepared for execution when capital market and tax reform provide suitable conditions for new investments.



Dr. E. Holmes, chairman of the Association of British Insecticide Manufacturers and Mr. F. W. Morris, Executive Director of Pest Control (UK) Limited of Cambridge and the new President of the British Agricultural Contractors Association, pose for a photograph at the BACA dinner at the Savoy Hotel on Wednesday, 9 December

Cotton Industry Research

THE vital part played by research in industry was stressed by Mr. N. G. McCulloch, chairman of the British Cotton Industry Research Association, at the recent annual meeting in Manchester. He mentioned that the council had been reconstituted to make it more fully representative of all sections of the industry.

The past year's work of the Institute was surveyed by its director, Dr. F. C. Toy. After dealing with the growing importance of mixed fibres in the industry, he spoke of further advances in sizing—supplying to the size box ungelatinised starch contained in a free-flowing cold aqueous suspension of special composition which had been patented.

On the 'finishing' side, great increase in contacts was reported: increase in works trials and particularly in stenter surveys. There had been further development in the mechanism of the pad-steam vat-dyeing process. The experimental raising machine was nearing completion and investigation into silk spinnning was beginning to yield results, especially with regard to long fibres.

Manx Lead & Zinc Mines

Work is to begin shortly at the old lead and zinc mines at Laxey, near Snaefell (Isle of Man), which have not been used for 45 years. A large quantity of plant is awaiting shipment from Liverpool, and roads are to be relaid in the valley leading to the mines. The first task will be to move some 80,000 tons of residue left by the old workings. The mines, now flooded, will have to be pumped out.

Recent Trends in Thermochemistry

by H. MACKLE, M.Sc., Ph.D., D.Phil.
(Department of Chemistry, The Queen's University of Belfast)

THERMOCHEMISTRY essentially began with the enunciation by Hess in 1840 of the law of constant heat summation. This law has for long been recognised as a special case of the first law of thermodynamics. Thermochemistry must therefore be formally considered as a branch of chemical thermodynamics. It is that branch which treats of the heat content changes associated with chemical reactions, and its ultimate aim is to provide experimental data from which may be calculated the heat of every possible chemical reaction.

The central importance in the general scheme of chemical thermodynamics of the heat content change, ΔH, associated with a chemical reaction is easy to appreciate. One only needs to remember that all thermodynamic functions may be derived from three fundamental quantities, of which the heat content, H, is one. The others are the internal energy, E, and the entropy, S.

It would not be proper, even if it were possible, to describe in a review like this the nature and uses of thermodynamic functions. There exist textbooks which perform that task very adequately. There is, however, an aspect of the general subject which needs brief comment in the present context because it contains an explanation of the high precision requirements for modern thermochemical data. It is this. One of the most important thermodynamic functions is the so-called free energy change ΔF, where $\Delta F = \Delta H - T\Delta S$. T is the absolute temperature, and H and S have already been defined. It is generally recognised that chemical affinity may be truly measured only in terms of ΔF, and ΔF is normally determined from equilibrium experiments.

There are, however, especially among organic compounds, a great number of substances for which values of the free energy change cannot be obtained in this way. The alternative is to determine the heat content and entropy terms separately. The latter may be got very accurately by means of spectroscopic data and the third law of thermodynamics. Accurate thermochemical determination of the former is therefore imperative, because in most cases the relative magnitudes and absolute accuracies of the values of ΔS and ΔH are such that the resulting uncertainty in ΔF is practically equal to the error in ΔH.

INDIRECT MEASUREMENT OF ΔH

It would obviously be impossible to determine ΔH for every conceivable chemical reaction. Fortunately, this is not necessary, for the same end may be accomplished indirectly by determining for each chemical substance its heat of formation from the elements in selected standard states. This is the scheme that has been used since thermochemistry began, and it is expressed precisely by the following relationship:—

if ΔH is the heat of the reaction $A + B \rightarrow C + D$, then $\Delta H = \Delta H_f(C) + \Delta D_f(D) - \Delta H_f(A) - \Delta H_f(B)$, where ΔH_f signifies the heat of formation of any particular substance from its elements in their standard states.

Clearly, therefore, the accurate determination of heats of formation is a thermochemical object of prime thermodynamical importance. It is also an important activity in its own right, for, provided the standard states are properly chosen, heats of formation give a quantitative measure of the chemical stability of molecules.

They are also indirectly useful in the elucidation of certain problems of molecular structure. For instance, steric effects may be detected because strain in a molecule causes its heat of formation to be less than that calculated. This effect is apparent in ethylene oxide, for example, where the deformation of the bond angles causes a decrease in the heat of formation of about 16 k.cal. per molecule.

Energy differences of *cis* and *trans* isomers may also be investigated. The heats of combustion in the *cis* and *trans* azo-benzenes differ by about 9 k.cal. per molecule.[1] This is mainly a steric effect because in the *cis* form the position of the benzene rings is

such that the conjugation energy is diminished relative to that of the *trans*.

Other effects which may be investigated by means of the heats of combustion are chain-branching effects, e.g., the heats of combustion of *n*-pentane, *iso*-pentane and *neo*-pentane in the gaseous state are 845.16, 843.24 and 840.40 k.cal. per molecule respectively.[2] Changes of hybridisation, and hyper-conjugation, also cause changes in the formation heats. For instance, the introduction of methyl groups into acetylene causes increased stability of the molecule by about 5 k.cal. per molecule per methyl group. This is not a simple effect, but hyper-conjugation is probably the main cause.

A common occurrence is that the observed heat of formation of a molecule is considerably greater than that calculated. This is nowadays attributed to resonance or π-bond delocalisation, and the difference between the observed and calculated heats of formation is called the resonance or delocalisation energy.

Finally, many heats of formation are necessary for the evaluation of the crystal energies of heteropolar compounds.

CHOICE OF STANDARD STATES

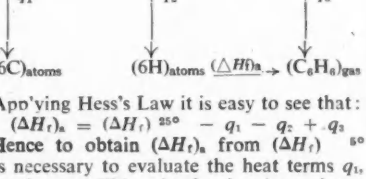
The conventional standard states of the elements are those forms which are most stable at room temperature and one atmosphere pressure. However, heats of formation based on such standard states are of little use as a measure of the comparative chemical stability of a series of compounds. This is because they are complicated by irrelevant condensed phase interactions, the integrated effect of which may vary considerably from one compound to another.

To obtain a true measure of relative chemical stability it is necessary to determine heats of formation of compounds in the gas phase from their elements, the chosen standard states of the latter being free gaseous atoms. Such heats of formation may be designated $(\Delta H_f)_a$. The problems connected with their measurement, and recent progress in resolving these problems, provide the subject matter for the remainder of this review.

VAPORISATION & SUBLIMATION HEATS

In setting up an internally consistent table of $(\Delta H_f)_a$ values it is necessary to select a standard temperature at which to carry out the actual calorimetric measurements. The most useful and convenient standard temperature is one at or near room temperature, and, following the Lewis-Randall convention for standard free energy values, the temperature chosen nowadays is 25°. Clearly, therefore, to obtain $(\Delta H_f)_a$ for any compound from $(\Delta H_f)^{25°}$ it is necessary to determine independently, and from a 25° origin, (a) the heat of vaporisation of the compound, (b) the heats of atomisation of its constituent elements.

The particular case of $(\Delta H_f)_a$ for benzene may be used to illustrate the point in terms of the following chemical scheme:—



Applying Hess's Law it is easy to see that:

$$(\Delta H_f)_a = (\Delta H_f)^{25°} - q_1 - q_2 + q_3$$

Hence to obtain $(\Delta H_f)_a$ from $(\Delta H_f)^{25°}$ it is necessary to evaluate the heat terms q_1, q_2 and q_3. This simply involves determining the heats of atomisation of graphite and hydrogen at 25° and the heat of vaporisation of benzene at the same temperature.

Until quite recently the accurate determination of heats of vaporisation and sublimation of chemical compounds has remained a comparatively neglected activity, and even today there are many important gaps in our knowledge of this field. This is probably because such data are not of great interest in themselves, but are only of general use when combined with other thermochemical data in a way already discussed. Quite often, therefore, those quantities must be estimated using such empirical rules as those of Walden and Trouton.

These estimation procedures are unsatisfactory. Fortunately, the vaporisation and sublimation heats are usually only small fractions of the required total heat terms, so the uncertainty in them is in general not disastrous. However, since this is not always so, it is clearly desirable to establish the entire subject on a firm and precise experimental basis, and there has been considerable activity in this direction during the past few years.

The methods used to determine heats of

vaporisation may be divided into three main groups:

(1) Those in which the heat required to vaporise a given mass of liquid is measured directly (evaporation methods).

(2) Those in which the heat given up by a given mass of vapour in condensing is measured directly (condensation methods).

(3) Indirect methods in which the heat of vaporisation is deduced from the variation of the vapour pressure with temperature, using the Clausius-Clapeyron equation. With certain exceptions[3] the modern emphasis is on this group and we shall confine our attention to it alone.

Details of the way in which vapour pressure/temperature coefficients may be used in the evaluation of vaporisation heats were set out fully by Thomson in a review article in 1946.[4] A fairly complete account of the various techniques employed up to that time for measurement of the vapour pressures is also given in the same article. Subsequently there have appeared several developments in technique of which perhaps the most noteworthy are those due to Balson[5] in 1947 and Bradley[6] in 1951.

Both these developments concern the measurement of small vapour pressures. The former is based upon the modification and marked improvement of a method originally due to Volmer[7] and later used by Newman and others.[8] The recoil force produced by jets of vapour effusing through holes is measured on a torsion balance.

The latter achieves precise vapour pressure data by measurements of effusion rates of vapour through a small hole in the platinum window of an effusion vessel. The rates are determined by the loss of weight recorded on a silica microbalance. Bradley and others[9] have lately used this method to determine the heat of vaporisation of KCl and the vapour pressures and lattice energies of various hydrogen bonded crystals.

Other interesting and recently developed methods of vapour pressure measurement have been devised by Dainton and Kimberley[10] and by Tickner and Lossing.[11] These are based respectively on the use of radioactive tracers and the mass spectrometer. Tschamler and Kohler[11a] have described a static vapour pressure method specially suitable for accurate work with small quantities of pure liquids.

A full and accurate survey of the available vapour pressure data for inorganic substances was given in 1935 by Kelley.[12] Subsequent data are recorded in various places. The best guides to recently published data are contained in the thermochemical review articles of Newton[13] and Stout.[14] The vaporisation heats of 88 organic compounds were listed in 1951 by O'Hara and Fahien[15] and values for many other organic compounds may also be traced from the papers of Newton and Stout (see above).*

During the past year Herrington and Martin[17] have measured the vapour pressures of pyridine and some of its homologues over the temperature ranges 50° to the boiling points. Other very recently published data include those of Edwards,[18] Drummond and Barrow,[19] Cook,[20] Johnston and others,[21] Oliver and others.[22] Inokuchi and coworkers have measured sublimation heats of some condensed polynuclear aromatic hydrocarbons.[23]

A paper of more general interest is that of Thomas.[24] This author has presented a generalised vapour pressure-temperature relation from which there evolves an apparently reliable method for comparison of vapour pressures.

Heats of Atomisation

Reliable values for the *heats of atomisation* of many elements are now available.[25] In some instances, however, the values are still uncertain. A notorious case is that of carbon. There exist at present three widely different experimentally determined sets of values for the heat of sublimation of graphite. There is the value L (C) = 170 k.cal. obtained from vapour pressure measurements at high temperature.[26] On the other hand a value of L (C) = 125 k.cal. has been derived by Herzberg[27] from spectroscopic studies of pre-dissociations in carbon monoxide. A different interpretation of the same evidence leads Gaydon[28] back to the 170 k.cal. value.

By the study of electron impact phenomena Hagstrum[29] arrives at L (C) = 137.5 k.cal. while McDowell and Warren[30] deduce L (C) = 120 k.cal. from similar impact work on methane and methyl cyanide. More recently Field[31] has considered the electron impact data and concludes that L (C) = 135

* The vapour pressures, Antoine Equation constants, and vaporisation heats of a large number of organic sulphur-containing molecules are available at the Thermodynamics section of the American Petroleum Institute, Bartlesville, Oklahoma. Some of the available data is already in print.[16]

k.cal. is the best value from such experiments. Kistiakowsky, Knight and Malin[32] give indirect reasons for favouring the value L (C) = 170 k.cal. Glockler[33] also favours the higher value. Pauling and Sheehan[34] support a value of L (C) = 140 k.cal. Very recently Chupka and Inghram[35] have reported a value of L (C) = 178.5 ± 10 k.cal.

It should be clear from the foregoing that the problem is by no means settled yet. A very good summary of the peculiar consequences of the uncertainty was written by Springall[36] in 1950.

Another quantity about which there has been much ambiguity is the heat of atomisation or dissociation of nitrogen. For determinations of $(\Delta H_f)_a$ values for nitrogen containing molecules this quantity must obviously be known. Here again three different values are available. Herzberg[37] and Douglas and Herzberg[37] favour 170 k.cal. from pre-dissociation spectroscopic studies. This value is supported by the electron impact work of Hagstrum.[29] Gaydon[28] arrives at a value of 225 k.cal. and Glockler[38] at an even higher one of 272 k.cal.

Kistiakowsky and co-workers[32] from detonation velocity measurements for mixtures of gases containing cyanogen, oxygen, nitrogen conclude that the value $D(N_2)$ = 170 k.cal. is too low. They suggest two possible values—225 k.cal. and 272 k.cal.—but eventually favour the lower of these. Pauling and Sheehan[34] support the low value of 170 k.cal. Thus, as for the sublimation heat of carbon, no clear-cut decision can be made at present regarding the heat of atomisation of nitrogen.

A third quantity which is not yet definitely fixed is the heat of atomisation of fluorine. A useful guide to the current literature on this problem, as well as on those already outlined, is due to Glockler.[39] The very recent work of Barrow[40] at Oxford is as yet unpublished.

A review of work on the vaporisation heats of group IV and group IVB elements, excluding carbon, has recently been given by Baughan.[41] It is not unlikely that, by analogy with carbon, the heat of atomisation of silicon, based on such vaporisation heat data, may be complicated by the participation of Si_2 molecules.

An unfortunate consequence of the above uncertainties in L(C), $D(N_2)$, $D(F_2)$, etc., is that definite *absolute* values of $(\Delta H_f)_a$ can be given at the present time for no organic compound and only for a restricted number of inorganic compounds. This seriously affects bond energy considerations. As Baughan[41] has pointed out, an energy error of x in the heat of atomisation of carbon implies a bond energy error of ¼ x in all single bonds to carbon, ½ x in all C-C bonds, etc. A full discussion of this aspect of the subject is not within the scope of the present review, and the problem is so complicated that a brief discussion would serve no useful purpose. The various theoretical and experimental questions involved have been considered in detail by many writers. Perhaps the most up-to-date and extensive critical reviews are those of Springall,[36] Glockler,[39] and Baughan.[41]

Fortunately, however, the general picture is not so depressing. It is only the absolute values of $(\Delta H_f)_a$ that are affected by the atomisation heat uncertainties. Differences between values of $(\Delta H_f)_a$ for different molecules may still be used as a quantitative measure of relative molecular stabilities. This is particularly true for isomeric molecules, and indeed for all cases where the uncertain atomisation heat terms disappear in a subtraction. Where this does not happen internally consistent stability comparisons may nevertheless be made, provided the original values chosen for the atomisation heats are maintained throughout. Furthermore, conclusions drawn from differences between calculated and observed heats of formation are not affected because the atomisation heats enter to the same extent in both quantities. This is most important from the viewpoint of resonance energy estimations.

EXPERIMENTAL DETERMINATION OF $(\triangle H_f)^{25°}$

As has already been demonstrated, the determination of $(\Delta H_f)^{25°}$ is the fundamental practical measurement of thermochemistry. All other measurements, such as those discussed in the previous section and additional ones like heats of solution and dilution, are ancillary to it. It is rarely possible, however, to determine heats of formation directly by measuring heats of syntheses from the elements. Indirect paths have to be employed, and (ΔH_f) computed by means of Hess's Law.

In organic chemistry the general procedure is simplified by the fact that the majority of organic compounds are combustible.

Their heats of formation are, therefore, most conveniently obtained from measurements of their heats of combustion. The only organic compounds which are not amenable to this procedure are those which contain exceptionally high proportions of oxygen, nitrogen or halogen and a small proportion of hydrogen.

In inorganic chemistry, on the other hand, the combustion process is of limited applicability since comparatively few inorganic substances react directly with oxygen. Roth and his co-workers have determined the heats of combustion of numerous elements, but, apart from this, reactions in solution have provided the major portion of the available thermochemical data relating to inorganic compounds. Recently the combustion technique has been adapted by Siemonsen[42] for determining the heats of formation of sulphides and chlorides, while the high temperature calorimeter originally developed by Biltz and others[43] in 1934 is a development which has not yet been exploited as fully as it might.[44] The general subject of the formation heats of simple inorganic compounds has very recently been thoroughly reviewed by Long[45] and it need not be pursued further here. The determination of the heats of formation or organic compounds via their heats of combustion is, on the other hand, a subject which, it seems to the writer, may usefully be discussed here at greater length.

HEATS OF COMBUSTION

There is a fundamental point concerning the derivation of heats of formation from heats of combustion that is sometimes not appreciated. It is this. Heats of combustion are determined by measuring the heat evolved when the substance is burned in oxygen under pressure in a bomb of definite volume. Thus what is measured is the internal energy change corresponding to that pressure. What is actually required, however, is the corresponding heat content change, ΔH, at a constant pressure of one atmosphere. As has already been implied, thermochemistry, by convention, concerns itself with reactions at this constant pressure, and not with reactions at constant volume. The bomb-calorimetric observations on combustion processes must therefore be subjected to an elaborate correction technique in order to obtain the true ΔH. A highly satisfactory correction procedure was evolved by Washburn[46] in 1933 and accepted in 1934 by the Commission Permanante de Thermochimie de la Union Internationale de Chimie.[47]

Furthermore, the computation of heats of formation of organic compounds from heats of combustion requires a precise knowledge of the heats of formation of H_2O and CO_2 in all cases, and of H_2SO_4, HCl, HNO_3 in those cases where the respective elements sulphur, chlorine or nitrogen occur in the original molecule. There is thus an interdependence of many of the values of heats of formation, and it is most important that values for key substances such as those just listed should be carefully and consistently selected.

When a change is made in any one of these basic values, corresponding changes should be made in all the values which depend upon it. There is a great need for a complete and internally consistent table of values and the achievement of this ideal is an important part of the now well-established project of Rossini and his co-workers.[25b] The position for hydrocarbons is now very satisfactory, but accurate values for the heats of formation of compounds containing nitrogen are still essentially limited to the work of Huffmann[48] on amino acids, and until very recently accurate values for compounds containing sulphur or halogens were virtually non-existent. Some of the reasons why this is so will appear shortly.

Typical combustion processes may be illustrated by those for benzoic acid and carbon disulphide. The simple reactions for these may be represented as follows:

$$C_7H_6O_2\ (\text{cryst.}) + 7.5\ O_2\ (\text{gas}) \rightarrow 7CO_2\ (\text{gas}) + 3\ H_2O\ (\text{liq.})$$

$$CS_2\ (\text{liq.}) + 4\ O_2\ (\text{gas}) \rightarrow CO_2\ (\text{gas}) + 2\ SO_3\ (\text{gas})$$

The reactions which take place in the bomb colorimeter are, however, a good deal more complex, and determination of the initial and final states requires great care. A true representation of the first of the above processes as it actually takes place would be something as follows:—

At 30 atmospheres pressure:

$$[C_7H_6O_2]\ \text{cryst.} + [aO_2 + bH_2O]\ \text{gas} + [c\ O_2 + d\ H_2O]\ \text{liq.} \rightarrow [(7 - e)\ CO_2 + f\ O_2 + g\ H_2O]\ \text{gas} + [e\ CO_2 + h\ H_2O + g\ O_2]\ \text{liq.}$$

Thus to obtain the required thermochemical value for the simple reaction at one

atmosphere the observed bomb reaction must be corrected, in addition to the fundamental correction already discussed, for the heats of solution of oxygen and carbon dioxide in water and the heat of vaporisation of water. The correction process is further complicated by the presence of some nitrogen in the oxygen used for the combustion. Because of this a correction must be made for the formation of a significant amount of nitric acid in accordance with the equation:

$$\tfrac{1}{2}\,N_2\,(gas) + \frac{5}{4}\,O_2\,(gas) + \tfrac{1}{2}\,H_2O\,(liq.) \rightarrow (HNO_3)\,aq.$$

The way in which all these additional corrections may be made has also been described by Washburn in the paper already referred to.[46] Very recently Sunner[49] and Sunner and Waddington[50] have shown how the Washburn corrections may be modified for the combustion products of sulphur containing molecules.

For a reaction to be amenable to accurate calorimetric study it should follow a single path, involve only stoichiometric compounds, and go to completion rapidly.* Combustion reactions fulfil very well the last of these conditions† because they are in the nature of explosion reactions. The entire amount of heat is therefore evolved in a very short period of time. The first condition is also generally fulfilled in combustion experiments, but the second depends on the purity of the starting material.

The problems connected with purification techniques and criteria of purity are manifold, and during the past ten years they have received considerable attention, especially those relating to calorimetric standard substances like benzoic acid.[52] Recently, the particular problem connected with the purification of sulphur-containing molecules have been thoroughly examined by the Thermodynamics Group of the American Petroleum Institute project.[90]

BOMB CALORIMETRY

The experimental technique of calorimetry in general has been developed to a high degree of precision during the past 25 years.[53] This is especially true of bomb calorimetry where the accuracy attainable nowadays is as a rule quite striking. Noteworthy among the more recently designed systems are those of Rossini,[54] Magnus and Becker,[55] and Casey and Fordham.[56] However, although all of these are highly successful for obtaining accurate heats of combustion data for C, H, O, and C, H, O, N compounds, the position for compounds containing sulphur or halogens has, until very recently indeed, remained unsatisfactory. This is reflected in the paucity of reliable data on such compounds.

The chief problem involved in combustion measurements on sulphur-containing molecules is that of devising equipment and techniques capable of yielding a thermodynamically-definable end-state in the bomb. When compounds containing sulphur are burned in oxygen the sulphur appears in the products of combustion in both its tetra- and hexavalent states. This mixture is unstable and impossible to define. It is the usual practice to direct the oxidation so that sulphuric acid alone is formed. A problem still remains, however, for the concentration of the acid formed varies throughout the bomb.

Since 1934 efforts have been made to burn sulphur-containing compounds in ways that would avoid such difficulties and yield accurate results. Becker and Roth[57] burned some sulphur compounds in a bomb to which 10 ml. of water had been added. After the combustion they made corrections for the difference in concentration of sulphuric acid in various parts of the bomb. It is probable, however, that this method does not give results of high accuracy, because equilibrium with respect to the solution of the bomb gases in the final solution is not established.

In 1935 Huffmann and Ellis[58] developed a method in which no liquid water is added to the bomb. They assumed that sulphuric acid was formed in the combustion process as a mist and condensed at a uniform concentration in various parts of the bomb. Recently Sunner[49] and Hubbard *et al.*[59] have shown that the concentration of sulphuric acid varies enough in different parts of the bomb to lead to serious errors.

The first really significant attempt to overcome these difficulties by using a comparatively large amount of water in a moving bomb is due to Sunner and co-workers.[60] Immediately after ignition the bomb is rotated, and in this way homogeneity in the final state of the combustion process is

* A successful method for studying slow reactions has recently been developed by Buzzell and Sturtevant.[51]

† Not all combustion reactions proceed readily to completion, and the extent to which they do should frequently be determined by analysis.

achieved. The method has many possible applications to other combustion studies, such as those relating to halogen-containing compounds, and it has been further developed very recently by Sunner, Hubbard, Katz, and Waddington at the US Bureau of Mines, Oklahoma.[61]

These authors have determined accurate values for the heats of formation of various thiols and sulphur derivatives of hydrocarbons. Their work is within the framework of the American Petroleum Institute Research 48A on 'The Production, Isolation and Purification of Sulphur Compounds and Measurement of their Properties.'

A rotating bomb calorimeter somewhat different in principle from those just referred to has been designed during the past two years by the present writer, and its construction is now nearing completion.[62] It is based on the aneroid or fluidless technique, originally tried by Dickinson and Osborne[63] and by White[64] some 35 to 40 years ago and since then comparatively neglected. It has a variety of advantages over the conventional systems which use well-stirred water or other fluid as the calorimetric medium. This appears to be particularly the case for a moving bomb system, where the design can be considerably simplified as a result.

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Chemical Stocks & Shares During 1953

by M. DUFFY

MOST heavy chemical company shares moved in favour of holders during 1953.

This will be seen from the share price table on the opposite page where the prices at the beginning of the year, the highests and lowests during the year and the end-year valuations are set out.

They consequently followed the trend of the entire industrial department of the London Stock Exchange. General factors mentioned in conjunction with this all-round improvement were the taxation concessions afforded to industry by the Budget in the spring and the reduction of the Bank Rate from 4 to 3½ per cent in the autumn.

Firm features were provided by Monsanto Chemicals 5s. Ordinary which finished at over four times their par value after having been dealt in up to nearly five times this amount, and by Distillers 4s. Ordinary which closed at over four times their nominal value.

Elsewhere, Johnson Matthey £1 Ordinary ended the year at nearly four times their nominal valuation, while Greef Chemical Holdings 5s. Ordinary and Ilford 5s. Ordinary both closed at nearly three times their respective par values.

Bowmans Chemicals 4s. Ordinary were noteworthy in that they closed the year at their par value.

Below Nominal Values

A few issues moved against the general trend by finishing the year under their nominal values.

These included Howards & Sons Ordinary, Pest Control 5s. Ordinary and United Indigo & Chemical 2s. Ordinary. At times during the year, however, they were around or over their respective par values.

Hickson & Welch 10s. Ordinary supplied a feature in this group as they remained under their nominal valuation for all of 1953.

In the Preference share section, end-year prices were mostly above the early-year valuations. It should be remembered, however, that dealings in Preference shares do not take place with the same regularity or freedom as business in Ordinary shares.

Firm features in the Preference share section were supplied by British Oxygen £1 6½ per cents and £1 5 per cents which both closed 1s. 6d. higher at 27s. and 21s. respectively, after having been transferred above these figures. Firmness was also evident for Fisons Chemicals £1 4 per cents which also finished about 1s. 6d. higher at 16s. 4½d.

Hickson & Welch £1 6 per cents were noteworthy in that they remained practically unaltered at 18s. 3d. for the entire year.

Most Regular Business

Business and inquiry during 1953 were most regular, among Ordinary shares, for Imperial Chemical Industries £1, British Oxygen £1, Courtaulds £1, Monsanto 5s., Staveley Coal and Iron £1 and United Molasses 10s. Orders in these were completed most business days and fair-sized trading was usually possible on the basis of a narrow difference between buying and selling prices.

Among shares which closed the year at several times their par values, Monsanto Chemicals 5s. Ordinary were noteworthy in that they issued their annual report in May of 1953, for the year ended 31 December, 1952. This showed net profit £261,000 odd against £911,000 odd for 1951. The 1952 dividend was reduced to 18½ against 22½ per cent. Around this time the shares stood in the region of 20s. 6d. From that time, however, their recovery to the year's highest of 24s. 4d., reached about the following October, was slow but fairly regular.

Bowmans Chemicals 4s. Ordinary, the only share in the attached list which finished the year exactly at their par value, were the centre of such a small volume of business and inquiry that it is doubtful whether this quotation can be regarded as having been adequately tested or established. In March of 1953 the company announced that they had made a loss for the year ending 31 October, 1952. They were consequently unable to recommend the payment of a dividend on Ordinary or Preference shares.

Name	Class	Price 1953 Early January	High	Low	Latest
Albright & Wilson	5/- Ord.	15/9	18/4½	14/10½	18/3
Do.	£1 5% Pref.	19/3	20/10½	19/-	20/7½
Bakelite	10/- Ord.	16/4½	24/6	16/-	23/3
Berk, F. W., & Co.	2/6 Ord.	5/9	5/10½	4/7½	5/7½
Do.	£1 4½% Pref.	16/9	18/-	16/-	18/-
Blythe, Wm., & Co.	3/- Ord.	9/6	6/3	4/6	6/3
Boake, Roberts, A.	5/- Ord.	11/6	11/6	7/6	10/-
Do.	£1 5% Pref.	16/6	17/-	16/-	17/-
Borax Consld.	£1 Defd.	37/4½	38/-	32/-	37/10½
Do.	£5 Pfd. Ord.	£5½	£5½	£5	£5½
Bowmans Chemicals	4/- Ord.	2/6	4/-	2/6	4/-
Do.	£1 5½% Pref.	12/6	12/6	10/-	10/-
British Glues & Chemicals	4/- Ord.	13/-	13/3	7/1½	10/10½
Do.	£1 8% Pref.	32/-	32/-	26/6	30/3
British Industrial Plastics	2/- Ord.	4/1½	5/-	3/7½	4/8½
Do.	2/- 10% Pref.	5/7½	5/9	5/-	5/9
British Oxygen	£1 Ord.	49/3	57/7½	48/3	55/3
Do.	£1 6½% Pref.	25/6	27/6	25/-	27/-
Do.	£1 5% 2nd Pref.	19/6	21/9	19/-	21/-
Do.	£1 4% 2nd Pref.	15/9	17/3	14/9	17/3
British Chrome & Chemicals	5/- Ord.	16/9	17/1½	15/6	16/6
British Xylonite	£1 Ord.	25/6	30/10½	22/6	27/-
Cooper, McDougall & Robertson	£1 Ord.	32/3	35/-	27/-	27/-
Do.	£1 7% Pref.	24/3	24/6	23/-	24/6
Courtaulds	£1 Ord.	38/6	50/6	36/-	50/6
Distillers	4/- Ord.	16/7½	18/4½	15/10½	17/7½
Do.	£1 6% Pref.	24/3	26/6	23/9	25/3
Do.	5% Loan Stock	103½	105¾	101½	104½
Fisons	£1 Ord.	30/6	39/9	30/-	39/4½
Do.	£1 4½% Pref.	17/3	18/9	17/-	18/7½
Do.	4½% Debs.	92	101½	90½	99½
Fisons Chemicals	£1 4% Pref.	15/-	16/6	14/-	16/4½
Greef Chemical Holdings	5/- Ord.	17/-	17/1½	13/9	14/4½
Do.	10/- 5½% Pref.	9/-	9/9	8/-	9/4½
*Hardman & Holden	5/- Ord.	18/6	10/4½	8/6	10/4½
Hickson & Welch	10/- Ord.	9/6	9/9	9/3	9/3
Do.	£1 6% Pref.	18/3	18/3	18/1½	18/3
Holliday, L. B.	£1 4½% Pref.	15/3	16/1½	15/3	16/1½
Howards & Sons	£1 Ord.	19/3	20/9	15/6	15/6
Do.	£1 5½% Pref.	17/6	17/6	16/3	16/9
Do.	5% Debs.	98	99	96½	97½
Imperial Chemical Industries	£1 Ord.	44/7½	51/3	42/3	50/6
Do.	£1 7% Pref.	26/7½	29/6	25/4½	27/10½
Ilford	5/- Ord.	13/4½	15/10½	12/4½	14/4½
Do.	£1 6% Pref.	22/-	24/-	21/6	23/9
Johnson, Matthey	£1 Ord.	67/3	79/3	64/4½	79/3
Laporte Industries	5/- Ord.	11/4½	13/-	9/6½	12/4½
Midland Tar Distillers	£1 Ord.	28/6	33/9	26/3	33/9
Monsanto Chemicals	5/- Ord.	23/6	24/4½	19/6	21/9
Do.	£1 3½% Pref.	15/-	16/-	14/6	15/10½
Do.	£1 4½% Pref.	18/4½	19/7½	17/10½	19/4½
Permutit	5/- Ord.	13/6	17/6	13/3	17/4½
Pest Control	5/- Ord.	4/3	5/10½	3/4½	4/7½
Do.	£1 6% Pref.	16/10½	17/9	16/10½	16/10½
Reichhold Chemicals	5/- Ord.	8/7½	8/7½	4/9	7/4½
*Staveley Coal & Iron	£1 Ord.	74/-	89/9	65/3	71/3
Unilever	£1 Ord.	45/3	58/-	45/3	55/9
United Molasses	10/- Ord.	29/-	34/3	26/6	31/3
United Indigo & Chemical	2/- Ord.	1/4½	2/-	1/1½	1/9
Yorkshire Dye and Chemical	5/- Ord.	7/6	8/6	6/3	8/6

* Ex-Bonus.

At that time the Ordinary were valued at the year's lowest, i.e., 2s. 6d. For the previous year they made a net profit of £27,300 odd and paid a dividend of 12½ per cent.

United Indigo and Chemical 2s. Ordinary, one of the shares which closed the year at under their par value announced their results, for the financial year ended 30 June, 1953, in the following September. Net profit was returned as £4,646 compared with £3,408. Ordinary shareholders benefited as their dividend was stepped up to 10 per cent from 7½.

Government Interest Urged

In a statement in Hobart recently, the Premier of Tasmania, Mr. Cosgrave, said that the Federal Government should have a financial interest in the oil industry. He said the fact that oil was waiting to be tapped should challenge the Government to make loan funds available to start what he described as 'Made in Australia' bores. Mr. Cosgrove added that it would be fitting if some of the rich profits from oil could find their way into the Federal Treasury.

Chemicals in Austria

Industry's Enormous Recovery

DURING the last decade a number of new chemical concerns was started in Austria. After the liberation in 1945, the chemical industry, helped by energetic support from abroad and the re-establishment of former business connections, was able to recover enormously, so that today the pre-war level has been reached in most sectors, and indeed in some cases exceeds it.

Furthermore, the production of various chemical products has been undertaken for the first time, e.g. carbon bisulphide, synthetic tannic materials, lead acetate, etc.

The development of the Austrian chemical industry has made it possible for Austria to compete in the individual fields of the world market of chemicals and to be thoroughly competitive in price.

High Quality Exports

Of special export importance is the nitro-chalk fertiliser which is manufactured by the Linzer Stickstoffwerke AG, and is exported in particularly large quantities to most European and overseas countries. The export of cellulose has also reached a remarkably high level owing to its quality.

Thanks to the ample Austrian power and water supplies the manufacture of hydrogen peroxide in the usual characteristic grades is so great that this branch of industry is able to place over 90 per cent of the total production at the disposal of the export trade.

The scarcity of raw materials allows the pharmaceutical industry to devote itself only partly to the export market. In spite of this, the industry is doing all it can to regain its place in the export trade, and to bring on the market again the pharmaceutical preparations which are as much in demand as ever.

Austria is in a position to export pigments, especially lead aluminium, soft lead, Vienna white, white lead and ultramarine, and soon will be able to place certain quantities of lithopone abroad. At the same time, there are good prospects for deliveries of Styrian Green Earth.

The highly developed Austrian production of opacifiers, enamels, as well as enamelled fancy articles and enamelled ceramics, enables considerable quantities to be exported.

Canada Distils Aromatics

CANADA'S chemical industry has taken another step forward in product self-sufficiency with first domestic distillation of aromatic petroleum solvents at the Petrolia, Ontario, plants of S. Nord Chemical Company. The aromatics are used in manufacture of a long list of products including paint and varnish, lacquer, degreasing solvents, resins used in paper coating and rubber maufacture.

It is planned to produce considerably more than the entire domestic market can absorb with substantial production going to promising markets in Michigan. Greatest production will be in benzene, toluene and xylene — all important chemicals often in short supply and used in dozens of manufacturing processes. Using coke-oven light oils from the steel industry, Nord Chemical will produce special nitration type (high-grade) benzene by the first continuous processed used in Canada.

Nord officials have plans ultimately to use large quantities of their own benzene, toluene and xylene in production of a range of coal-tar derivatives basic to the dye, rubber, perfume and other industries, and never produced before in Canada.

Aluminium Prices Increased

BECAUSE of the increase in the market price of virgin aluminium ingot from £150 to £156 a ton, and increases in manufacturing costs not previously reflected in prices, the British Aluminium Co., Ltd., have made increases in their selling prices as follow:—

Rolled Products:—Pure aluminium and BA.60 alloy, 1½d. a lb.; BA.21, 25, 27 and 28, 1d. a lb.; strong alloys, 1d. a lb.; box-making and capping quality, 1½d. a lb.; utility grade —pure aluminium and BA.60 alloy, 1d. a lb.; corrugated sheet, equivalent of 1d. a lb.; foil stock, £8 a ton.

Extrusions and tubing, 1d. a lb.; slugs, 1½lb. a lb.; paste, 1d. a lb.

Details of increased prices for virgin aluminium and aluminium alloy ingots, slabs, billets, hardeners and granulated powder are available on application to the company at Norfolk House, St. James's Square, London, S.W.1.

A Founder of Physical Chemistry

by DR. GABRIELE RABEL

WILHELM OSTWALD, the famous German scientist who created—together with Svante Arrhenius and J. H. van t'Hoff—the science of physical chemistry, was born just over 100 years ago. After his death in 1932 his daughter Grete transformed his estate 'Energie,' near Leipzig, into 'Ostwald Archives.'

On 2 September last, the University of Leipzig, now in the Soviet Zone, commemorated the hundredth anniversay of the birth of one of her greatest sons. The celebration began in the usual manner with the usual eulogies, but then something very unusual and very dramatic happened. A 'messenger' from the Government appeared on the stage and proclaimed that from now on the Academy of Sciences would enter into possession of the whole Ostwald estate, take into their special care and protection the Archives, including their keeper, and, realising the fervent dream of Ostwald's last years, begin immediately the erection of an Institute for Colour and Dye Research.

A Readable Biography

Ostwald's youngest son Otto, a chemist and engineer who had for many years been his father's collaborator, is to be the first director of the new institute. Ostwald's eldest son Wolfgang, the colloid chemist, died in 1943. His daughter Grete, though affected with polyarthritis since the first world war and almost wholly paralysed, has gallantly contrived to celebrate her father's anniversary by writing his biography. This book, eminently readable and wholly sincere, has just appeared, and the facts mentioned in the present article are mostly drawn from it.

Ostwald's birthplace was Riga, his university Dorpat, both places situated in the Baltic Provinces. These were politically under Russian dominion, but the aristocracy and the intelligentsia were German in language and sentiment.

C. Schmidt and J. Lemberg taught young Ostwald chemistry, while Oettingen taught him not only physics but also musical harmony. His first lectures, delivered to a ladies' circle, dealt with Beethoven's sonatas.

In 1881 he was called to fill a chair for chemistry at the Riga Polytechnicum, and then and there the never ceasing stream of chemical text books began to flow from his desk.

The appearance of Arrhenius' doctor's thesis in 1884 provoked from Ostwald this comment: 'I had to realise that the great problem of chemical affinities, to which I intended to devote all my life, had been solved by this young man in a much more comprehensive way than I had tackled it.' Immediately Ostwald published a paper to 'advertise' his rival's achievement and even travelled to Sweden to meet him. A lifelong close friendship ensued.

British Association Invitation

In 1887 Ostwald became professor in Leipzig. A new institute built for him was soon overcrowded with eager students who came from all corners of the world. The first assistants included Arrhenius and Nernst. His inaugural lecture was on 'Energy and its Transformations.' In the same year he founded, together with van t'Hoff, the first organ for their new science, the 'Zeitschrift für Physikalische Chemie.' This caused a considerable stir. It also led to an invitation for the British Association meeting at Leeds in 1890 to discuss the theory of solutions.

'I assume,' wrote Ostwald, 'that our English colleagues desired to enlighten us about our misconceptions and then send us home hoping that we would mend our ways. . . . Indeed, on Sunday it looked as if we were scientific corpses. But on Monday the scales were turned. . . . We owe it partly to the intervention of William Ramsay that our otherwise so conservative cousins quickly adopted the new doctrine. Two years later in Edinburgh the battle was over. I found myself placed on the platform between Lord Kelvin and Hemholtz, feeling like a mouse between two lions.'

Another battle started at a congress in Lübeck in 1895, when Ostwald spoke on 'The Defeat of Scientific Materialism.' He expounded that matter is nothing but a relatively stable combination of various energies, that energy itself is the only permanent substance and that all phenomena ought

to be described in terms of energy. Planck, among others, opposed the 'New Energetics' and stated that Rankine's 'old energetics' (oddly born in the same year as Ostwald, 1853) had proved sterile.

Ostwald's restless mind—the mind of a true 'romantic,' according to his own classification—soon burst the boundaries of physical chemistry. From 1900 he lectured on 'Naturphilosophie' and issued a journal which became an organ for the Positivists—the 'Annalen der Naturphilisophie.'

In a Faraday Lecture in London he tried to show that all Dalton's results could be obtained without hypothetical atoms. Later he welcomed the advent of the Quantum Theory as a combination of atomism with energetics.

In 1906, after a half-year as exchange professor in Harvard University, he 'exchanged the life of a Leipzig don against that of a practical idealist,' retiring to his country seat 'Energie.' In 1909 he was awarded the Nobel Prize for his early studies on catalysis.

Ostwald's last years were entirely devoted to what he considered his most important work, a systematic study of the harmony of colours. By thousands of measurements and experiments he constructed a colour atlas in which every shade is described by three variables (tint, black and white) and can be produced at will. His aim was that a symphony of changing lights and colours may be written down as is the score of a musical symphony. Here his lifelong passionate devotion to science and art merged in one. He was enchanted by the beauty of synthetically composed harmonies. And eastern Germany is now going to build the Colour Research Institute which he envisaged.

African Mineral Resources

Africa is destined to become one of the most important producers of minerals in the world, said Mr. Wishart Philips, a Johannesburg industrialist, in a recent speech on the commercial possibilities in the Belgian Congo and Angola. The continent already provided three-quarters of the world's cobalt and uranium, and 70 per cent of the world's industrial diamonds. It had other even more fantastic resources, and its exploitation would create new markets which could be supplied by South African industries.

New Type Vacuum Pump

American Improvement Described

UNIVERSITY of Wisconsin physicists have devised a new type of vacuum pump which eliminates the organic vapours that restrict the usefulness of conventional types of oil vacuum pumps for some purposes. The new pump was described by Professor R. G. Herb, Ajay Divatia, and Mr. Robert Davis to physicists attending the recent annual meeting of the American Physical Society in Chicago.

Titanium Wire Used

Pumping speeds of more than 6,000 litres a second for oxygen, nitrogen and hydrogen were claimed for the new type pump. The pump uses wire made of titanium, which because it is extremely difficult to obtain in pure form, became available commercially only recently.

The pump is so designed that titanium wire is continuously evaporated in a vacuum chamber by contact with a carbon rod heated electrically. The titanium condenses on the chamber walls and molecules of atmospheric gases either combine with the titanium molecules to form stable compounds or, in the case of inert helium, argon, and other noble gases, are ionised and driven to the wall of the vacuum chamber by electrical fields, where they are trapped and buried by the condensing titanium.

Continuous operation of the pump is achieved by automatic motion of the titanium wire into contact with the hot carbon rod as it progressively vaporises.

The titanium pump is an improvement over the conventional oil pump because there is no possibility of oil or other organic compounds diffusing throughout the vacuum chamber. These compounds are particularly troublesome in research conducted in nuclear physics or with X-rays because the organic materials oxidise and form thin layers of carbon over the nuclear particle or X-ray targets, destroying their effectiveness.

The pump was developed to render more precise the experimentation which will be conducted when a new electrostatic generator being built at Wisconsin goes into operation. 'The pump shows considerable promise, but further development is needed before it will be dependable for long, trouble-free service,' said Professor Herb.

The Nickel Industry in 1953

Worldwide Demand Remains High

DEMAND for nickel throughout the free world in 1953 continued at a high level, with defence needs again restricting the amount of the metal available for civilian applications, Dr. John F. Thompson, chairman of the board of directors of The International Nickel Company of Canada Ltd. has stated in a year-end review of the nickel industry.

It is estimated that almost half of the production of the free world during the year was allocated for its military, atomic energy and stockpile requirements. Over 65 per cent of this 1953 output went to the United States for both defence and civilian purposes.

Production of nickel by the free world is estimated to have been 340,000,000 lb. last year, an increase of about 20,000,000 lb. over 1952, and 50,000,000 lb. over 1951. Canadian output is estimated to have reached 285,000,000 lb. in 1953, or about 85 per cent of the free world's total production.

Governmental restrictions on the allocation and end uses of nickel in various countries, which had been in effect for several years, were substantially relaxed in 1953. The relaxation of controls occurred as a result of the progressive decrease in military demands, commencing in mid-year, and the maintenance of a constantly high level of nickel production throughout the year.

Expansion Programmes

During 1953, the nickel industry continued its extensive search for new nickel deposits. In Canada, particularly, this programme has been on a scale far surpassing anything previously undertaken. The established producers of nickel have underway expansion programmes which, with the output of new companies in the field, should further increase the overall supply of nickel available to the free world.

Applications of nickel in 1953 were necessarily influenced by governmental restrictions prevailing during most of the year. The steel industries of the United States, the United Kingdom and Canada, in the production of stainless steels, engineering alloy steels, and jet engine alloys, were again the largest consumers of nickel.

As nickel becomes available in larger quantities for civilian uses, manufacturers of products for the architectural, appliance and utensil fields are expected to return to the nickel-containing types of stainless steels because of their good fabricating characteristics and other outstanding properties.

The amount of primary nickel consumed in the broad range of engineering alloy steels, which find widespread applications in both military and civilian equipment, is estimated to have been slightly larger than in the previous year. These steels continued to provide the high level performance needed in essential parts of aircraft engines, trucks, power generation machinery, railroad transportation equipment, earth moving and road building machinery and farm equipment such as tractors.

Defence Requirements

Production of high nickel alloys in the United States and the United Kingdom continued to expand in response to defence requirements. In these countries and Canada there were broad applications of the Nimonic and Inconel series of nickel-chromium alloys in components of both jet and reciprocating aircraft engines. Monel, a nickel-copper alloy, and Inconel again found employment in industry in approved uses, such as in the chemical handling field, where resistance to corrosion or heat and high strength are needed. Monel was required in oil well drilling equipment and as corrosion-resistant sheathing to protect off-shore drilling platforms. Incoloy found continued demand as heat-resisting alloy furnace tubing in the production of ethylene in the petrochemical field.

In the distribution of nickel which, during most of the year, was under government allocations, the nickel-plating industry did not receive a sufficient quantity of the metal to maintain pre-Korean War practice in respect to the thickness of coatings. The nickel supply for plating became somewhat better by the latter part of the year through increased allocations made both before and after government nickel controls were relaxed.

Manufacturers of nickel silver, since the relaxation of controls, are returning to the

production of the 18 per cent nickel-containing grades of this copper-nickel-zinc alloy to the extent permitted by supplies of nickel. Prior thereto, the percentage of nickel allowable, even in permitted uses of this alloy, had been radically reduced and many applications prohibited entirely.

Production of cupro-nickel alloys during the year was at about the same level as in 1952, although demands for both the 10 per cent and 30 per cent nickel grades showed an increase, particularly in recent months.

Nickel-chromium alloy castings are essential in heat-treating furnace equipment for both the maintenance of existing facilities and the installation of new units. A significant amount of nickel was employed in this field during 1953.

Ni-Hard, the abrasion-resisting nickel-chromium cast iron, continued to serve in essential applications for grinding balls in the mining, cement and paint industries. Ni-Resist, the corrosion-resisting nickel alloy cast iron, was again used in components of industrial equipment where resistance to corrosion, heat and wear is required.

The magnetostrictive property of nickel continued to arouse interest in widening fields. One of the first applications of this interesting characteristic of nickel was in depth-finding instruments on ships, which was later expanded to 'Sonal,' an ultrasonic submarine detecting device. During the year several new fields employing this principle have been developed, including such diversified applications as an electronic machine tool capable of cutting intricate shapes in the hardest materials, oil well drill bits which markedly increase drilling rates in hard rock, a burglar alarm, and a device for detecting whales and schools of fish.

Ethyl Alcohol Prices

Reductions Announced This Week

THE Distillers Company Ltd. (Industrial Alcohol Division) announced this week that the prices of plain British spirits have been reduced.

A reduction of 2d. per proof gal. has been made on all sizes of delivery of ethyl alcohol 95 per cent (66.0 o.p.). The basic schedule for tank wagon deliveries of this grade now ranges from 2s. 11½d. per p.g. for totals of 2,500-9,999 p.g. per annum, to 2s. 9d. per p.g. for totals over 300,000 p.g. per annum. The price of absolute alcohol re-rectified 99.7 per cent (75 o.p.) remains at 12s. 6d. per bulk gal.

The effective reduction on all other grades of plain British spirit is 1d. per p.g. as it has been found necessary to increase by 1d. the premiums above basic schedule price for these grades. The premiums of 1d. and 3d. per p.g. for deliveries in 40-gal. drums and in 10-gal. containers respectively remain unchanged.

The Methylating Co., Ltd., anounced reductions in the prices of methylated spirits and finish. For tank wagon deliveries (min. 500 gal.) the new prices for IMS range from 4s. 8d. per bulk gal. for 61 o.p. to 5s. 5d. per bulk gal. for 74 o.p.; for IMS (toilet quality), from 5s. 2½d. per b.g. for 61 o.p. to 5s. 10d. per b.g. for 74 o.p.; and for pyridinised IMS from 4s. 11d. per b.g. for 61 o.p. to 5s. 1d. per b.g. for 66 o.p.

Premiums for delivery in packages from 30 gal. upwards range from 7½d. down to 1½d. per b.g. according to grade, strength and quantity. Prices of all grades for deliveries of less than 30 gal. are unchanged.

Prices for IMS (D.M.5 quality) and IMS ('Q' grade 68 o.p.) are 4½d. and 1s. 5d. respectively above IMS (toilet quality) prices. Prices for methylated resin finish and methylated shellac finish are 3d. and 1s. per gal. above pyridinised IMS prices.

'Re-Birth' in West Germany

WEST Germany's badly-bombed chemical industry is experiencing rebirth of its inventiveness and is making progress, according to Dr. Th. Merz, Canadian representative of the Farbenfabriken Bayer Leverkusen when speaking in Montreal recently.

He told a group of representatives of the Montreal dye-stuffs industry that Germany's chemical production had again achieved efficiency, but was faced with obligations to provide decent housing and a fair standard of living for its employees.

Dr. Merz said Germany's chemical industry was keenly interested in Canadian development, especially in the field of raw materials which could be used in Germany. He added that there were very friendly relations between the business people of the two countries.

The Chemist's Bookshelf

VON ATOM ZUM UNIVERSUM (Einführung in die Kosmologie). By Hans Vogel. Hans Carl, Nürnberg. 1953. Pp. 292. DM. 10.80 (boards), DM. 12.80 (cloth).

This volume sets out to acquaint the layman with the discoveries of modern science and to show him how these effect man's 'Weltanschauung.' Physics (atoms, radiations, fundamental particles, quantum theory, periodic table, uncertainty principle, etc.), geophysics, astronomy, biology and the philosophical implications of the findings of these sciences are all compressed into less than 300 pages. As might be expected, the treatment is very superficial. Technical terms are often used without an explanation of their meaning and the philosophical sections sometimes become unduly fanciful. The layman who reads German will find this book easy to read—it will give him a broad and rather vague picture of science without calling for any great mental effort. However, it is not recommended to the critical reader.—J.C.P.S.

ORGANIC SYNTHESES. Vol. 33. Edited by C. C. Price. John Wiley & Sons Inc., New York, Chapman & Hall Ltd., London. Pp. 115. 28s.

The latest edition of the 'annual publication of satisfactory methods for the preparation of organic chemicals' lists 41 compounds in alphabetical order. In fact, however, the preparation of many more may be carried out with the aid of instructions given in the text, since the methods quoted often have a general application. Thus the preparation of 3-acetamido-2-butanone by the action of acetic anhydride on alanine may be applied to the production of ketones from other amino acids and anhydrides while *m*-dioxanes are prepared from a variety of aryl olefines. In addition, one preparation may involve the formation of several intermediates, thus increasing the number of compounds described. The preparation of 3-methyl-coumarone, for instance, involves ethyl 3-methyl-coumarilate and the parent 3-methyl-coumarilic acid.

The increasing concentration of the organic chemist upon substances of natural origin finds a reflection in the present series by the provision of methods for preparing biologically significant intermediates. As an example 1,4-naphthaquinone is a structure which appears in antibiotics, plant pigments and vitamins, while nicotinonitrile is too familiar in this sense to require comment.

One of the most interesting reactions is the ring scission and dehydrochlorination of tetrahydrofurfuryl chloride with sodamide in liquid ammonia to yield 4-pentyn-1-ol, a substance closely allied to 'Oblivon', the acetylenic alcohol manufactured as a substitute for barbiturates.

A single gummed leaf is supplied with this issue for insertion opposite p. 95 of Vol. 32. It contains a warning concerning the handling of the sodium salt of nitromalonaldehyde which is thermally unstable and sensitive to shock.

On p. 84 the word 'acid' has been omitted from the description of the preparation of stearone from stearic acid; fortunately this makes no alteration in the sense of the text.

The index at the end of the book is a cumulative one covering volumes 30-33, and there are the usual bibliographies at the end of each preparation, many containing over a dozen references. J.R.M.

A TEXTBOOK OF PURE AND APPLIED CHEMISTRY. By James E. Garside and R. F. Phillips. Sir Isaac Pitman & Sons, London. 1953. Pp. 1044. 25s.

In 1044 pages, the two authors have tried to supply a textbook on the entire field of pure and applied chemistry. Mainly a compendium for students of chemistry, metallurgy, metal finishing, fuel technology, chemical engineering, and ceramic technology, it is intended to serve also as a general review of the vast and important field of

pure and applied chemistry for the technician in every section of industry. The technical or science student is apt to become more and more specialised and as Einstein once said, 'A specialist is a man who knows more and more about less and less until finally he knows everything about nothing.' It is therefore important that an overall picture of the entire chemical field should be given so that specialist students can see their own position in its context, and in relation to everyday life.

The book is split into three sections: General Theoretical and Physical Chemistry, Systematic Inorganic Chemistry, and Organic Chemistry. Practical application of theoretical knowledge is given in connection with each section. In view of the range covered it is not to be wondered at that no aspect can be dealt with very fully, but on the whole the authors have succeeded in what they set out to do, i.e., to give an overall picture.—FELIX SINGER.

'THE ALKALOIDS—CHEMISTRY & PHYSIOLOGY.' Vol. III. Edited by R. H. Mauske and H. L. Holmes. Academic Books Ltd., London. 1953. Pp. viii + 422. $11.00.

In the present volume the chemistry of the alkaloids having quinoline, quinazoline, imidazole and β-phenylethylamine structures, and of the lupin, solanum, veratrum, ephedra, and ipecac alkaloids is reviewed by a number of authors of established reputations. Most of the chapters are compendious rather than inspired, although their value to workers in the fields under review can hardly be exaggerated. However, Richard B. Turner and R. B. Woodward have surveyed the chemical transformations, stereochemistry, and biogenetic relationships of cinchona alkaloids in a chapter that is a masterpiece of condensation and clarity. As would be expected in any work by these distinguished authors, extensive use is made of modern theoretical concepts in their account. This chapter should have a wide appeal to all chemists, and can be confidently recommended to any student wishing to learn something of the methods and possibilities of alkaloid chemistry.

For most of the alkaloids the physiological action is not described in any detail. However, the effects of mescaline, which causes colour hallucinations, are described in an interesting paragraph (p. 333): 'Ordinary objects appear to be marvellous. Sounds and music are "seen" in colour. In comparison, the impressions of every day life seem pale and inert. Colour symphonies and new, unknown colours of unimaginable beauty and brilliancy are perceived.' This drug, obtained from dried heads of the cactus, has been used by Mexican Indians in various religious rites.—J. T. EDWARD.

INITIATION A LA SYNTHESE ORGANIQUE. By J. Lenoir. Presses Documentaires, Paris. 1953. Pp. 293. Limp cardboard cover. No price given.

This book was written for the 'Centre Technique D'Enseignement Ouvrier' and is founded on considerable teaching experience in this institution. It will be of special interest to those who train technicians for the chemical industry, as it provides an elementary course in organic chemistry which is entirely based on compounds and processes used industrially. The course is divided into three sections: General (formulæ, functional groups, nomenclature and other fundamental concepts), Primary Materials (the preparation, properties and uses of most industrial starting materials) and Principles of Organic Synthesis (dealing with the more important reactions used in industry—intermediate and final products are also discussed). The material is very well presented and those who are in sympathy with M. Lenoir's teaching scheme will find this a very useful book. It can be used as an introduction to two more advanced texts by the same author.—J.C.P.S.

Poisons List Changes

Statutory Instruments giving effect to recommendations made to the Secretary of State by the Poisons Board are being prepared. It is proposed to make the following changes in the Poisons List, and in the Schedules to the Poisons Rules:—Tris-(2-chloroethyl)-amine will be added to the series of nitrogen mustard compounds already included in Part I of the Poisons List, and in the First and Fourth Schedules to the Poisons Rules. Rat and mouse preparations containing not more than 0.1 per cent sodium nitrite will be exempted from the requirements of the Pharmacy and Poisons Act and the Poisons Rules.

HOME

Teleprinter Facilities

New Metals & Chemicals (Sales) Ltd. announce that they have installed International Teleprinter facilities, their Telex number being: London 8016.

Employment in Chemical Industry

Statistics given in the *Ministry of Labour Gazette* for December, 1953, show that 497,500 people were employed in the chemical and allied trades at the end of October, 1953, as compared with 486,000 at the end of October, 1952. The largest proportion—217,800—were in the chemical and dye trades.

Exports to Canada

Of importance to exporters of goods to Canada is the announcement that in order to facilitate the more rapid compilation of trade statistics, the Canadian Department of National Revenue has agreed to furnish the Dominion Bureau of Statistics with a copy of each customs import entry for use and retention by the bureau. As from 1 April next, import entries will be required to be presented at Customs in triplicate at ports and quadruplicate at outports, in place of in duplicate and triplicate as at present.

Lectures on Atomic Energy

The Atomic Scientists' Association, in collaboration with the University of London, are organising a series of six public lectures on various aspects of atomic energy, given by leading authorities in the subject. These will be held in the Beveridge Hall, Senate House, London, W.C.1, on Wednesdays at 7 p.m., commencing 20 January. The fee for the course is 10s., or 5s. for members of the Atomic Scientists' Association. Applications for tickets should be addressed to the Cashier, University of London, Senate House; single tickets, price 2s., may be obtained at the door.

West Country Agent

Steele & Cowlishaw Ltd., Cooper Street, Hanley, Stoke-on-Trent, manufacturers of ball and pebble mills, and mixing machinery for the paint, chemical and allied industries, have appointed Mr. R. R. Britton, of Memorial Road, Hanham, Bristol, as their Bristol and west country agent.

Export of Petrol Products

The export of petroleum products from the UK is now at a higher rate than ever before. A deal which will bring £7,500,000 in foreign currency to the UK has recently been concluded between the Esso Petroleum Co. Ltd. and the Danish Esso Co., whereby during the next year the latter will buy from the British company approximately 700,000 tons of petroleum products.

Apprenticeship Anomaly Removed

Lever Brothers (Port Sunlight) Ltd. and the trade union concerned have reached agreement on an adjustment of the age of entry into craft apprenticeship so that boys make take the examination for the General Certificate of Education before starting work. Hitherto, boys born in certain months were virtually prevented from sitting for the certificate because the school calendar conflicted with the maximum age for entry into apprenticeship.

Laboratories Moved

On 31 December, 1953, the Reading laboratory of Thomas McLachlan and Partners, public analysts and agricultural chemists, moved to the former County Police Station, Abbey Street, Reading. The old County Police Station is the second building south of the Abbey Gateway and the laboratory faces the main entrance gate. The address is Thomas McLachlan and Partners, Abbey Street, Reading. Telephone: Reading 4501. Telegraphic address: Macslab, Reading. The London laboratory remains as at present at Hanway Place, W.1.

Anglo-Iranian Oil Co. Ltd.

Anglo-Iranian Oil Co. Ltd. state that there is no truth in reports that, in the recent informal talks in London between representatives of several oil companies, the setting-up of an internationally-owned marketing company to sell Persian oil, or other measures in respect of the Persian oil situation, were even tentatively agreed. As announced by Anglo-Iranian at the time, the sole purpose of these talks was to explore the problems involved if and when Persian oil should again flow into its traditional eastern hemisphere markets. The talks were exploratory only.

OVERSEAS

Bengal Oil Exploration

Negotiations have been started by the Indian Government with the Standard Vacuum Co. to conduct ground explorations for oil in the Bengal Basin on a two-year agreement at a cost of approximately £1,500,000.

Canadian Oil Pipeline

A message from Toronto states that the laying of an additional 600 miles of oil pipeline parallel with portions of the present Edmonton-Superior crude oil carrier is expected to raise capacity from the present potential daily throughput of 170,000 barrels to 200,000 barrels.

Record Oil Drilling in USA

According to the Petroleum Information Bureau, it is estimated that about 50,000 boreholes were sunk in the United States of America last year in search of oil. This establishes a record, passing the 1952 total by just over 500. Most of these are producing wells drilled in proven areas, but more than 20 per cent are holes sunk in areas where oil is not yet being produced.

Australia's Petrol Imports

During the past financial year Australia imported petroleum products costing £A72,000,000. About £A12,000,000 worth was bought direct from dollar areas and paid for in dollars. Dollars were also paid for considerable quantities of petroleum products from the sterling areas because some of the oil companies operating there had their headquarters in North America.

Petroleum Course in Melbourne

Australia's first full-time petroleum engineering course will open this year in Melbourne Technical College. The course, which will take five years, will cover all phases of oil production from well-drillings to the refining of petrol and by-products. The Principal of the College said recently that if Australia's oil industry expanded, as seemed likely, technical men would have to be brought from overseas if they could not be supplied locally. The decision to institute a course follows the recent discovery of oil in Exmouth Gulf, in Western Australia, and the possibility of more drilling and an expanded search for oil. Six large refineries are at present being built in Australia.

To Increase Production

Through Claritude Limited we have learned that their principals, the Saint-Gobain Company of France, are about to increase their production of maleic acid and maleic anhydride. It is hoped that the new plant will be functioning towards the end of June this year.

Spanish Nitrate Factory

A new factory for the production of sodium nitrate is to be put up by the Spanish Explosives Union Company at its Galdacano works. The plant will cost more than 8,000,000 pesetas (£71,000), of which 5,000,000 pesetas will be spent on importing foreign machinery. It is expected that the output of the new factory will amount to 2,500 tons a year.

Hungary to Grow Pyrethrum

Large-scale growing of pyrethrum for insecticidal purposes has begun on the experimental farm of Akál, near Lake Balaton, in Hungary. Hitherto unproductive soil in the district of Udver has been found suitable for cultivation of pyrethrum, previously grown in Hungary only as a garden flower.

Pakistan Newsprint

The United Nations Technical Assistance Administration has decided to help Pakistan in manufacturing newsprint from local material, and has selected a Swiss expert to advise the Pakistan Government on the subject. The project is aimed at the development of the newsprint industry in East Pakistan, where tropical soft woods, including superior species, are found in abundance.

The First of its Kind

The B. A. Shawinigan Ltd. plant, the first of its kind in the world to produce phenol and acetone from cumene, at Varennes near Montreal, is now partly operational. Among the by-products are α-methyl styrene and cumene hydroperoxide. α-Methyl styrene is an interesting monomer with outlets in injection moulding and surface coatings. Cumene hydroperoxide is a catalyst which will be available shortly. This plant is linked with this country through Shawinigan Ltd., of Marlow House, Lloyd's Avenue, London, E.C.3.

· PERSONAL ·

Among the Knights Bachelor in the New Year's Honours List (p. 130) MR. C. G. GRAHAM HAYMAN, chairman since April, 1953, of the management committee of The Distillers Co. Ltd., which controls all the activities of the DCL organisation. For the past 30 years Mr. Hayman has been very closely connected with the distilling and industrial interests of DCL, and he became a director of the parent company in 1936.

Before assuming his present responsibilities, Mr. Hayman was chairman of the DCL Industrial Group executive committee, which has carried through the company's extensive programme of post-war industrial expansion. He represents DCL on the boards of National Chemical Products Ltd. (South Africa) and of CSR Chemicals Pty. Ltd. (Australia), whose new chemicals plant at Rhodes, NSW, was recently completed. He is in addition chairman of the British Tyre and Rubber Co. Ltd.

In December, 1951, he was a member of the British delegation to the first International Conference of Manufacturers, organised by ECA and held in New York to examine problems of European and American production and productivity. From October, 1950, to October, 1953, he was chairman of the Association of British Chemical Manufacturers.

The Mond Nickel Fellowships Committee has announced the following awards for 1953:—

MR. J. E. BENSON (Metropolitan-Vickers Electrical Company, Manchester) to study the techniques and interpretation of results of non-destructive testing of metal components in the UK, on the Continent and in the USA and Canada.

MR. K. BLACKBURN (Dorman Long & Co., Redcar) to study hot-metal basic open-hearth practice in Great Britain, on the Continent and in the USA and Canada, with particular reference to mixed furnace operation, refractories, instrumentation and pit-side practice.

MR. N. B. PRATT (Broken Hill Proprietary Company, Newcastle, NSW) to study the technical and economic aspects of recent advances in the erection and operation of integrated iron and steelworks in Great Britain, on the Continent and in the USA and Canada.

MR. GILBERT DODD, who is named as general manager of the new Monsanto subsidiary, Monsanto Plastics Ltd., is well known in the plastics industry both at home and abroad. He joined Monsanto's sales division in 1939. In 1949 he was appointed manager of the plastics department, and in 1953 became general manager of Monsanto (Soil Conditioners) Ltd., which company is responsible for the retail marketing of Krilium soil conditioner.

Among the directors of the new company, MR. P. A. SINGLETON is managing director, and MR. D. R. MACKIE commercial director, of Monsanto Chemicals Ltd.

MR. SIDNEY J. VAN DEN BERGH has been appointed a vice-chairman of Unilever NV.

MR. ROBERT CRICHTON, managing director of Scottish Oils Ltd., has indicated his pending retirement, to take effect from the end of March 1954. One of the best known men in the oil trade in this country, Mr. Crichton was trained as a mining engineer and became engineer and general mining manager to Scottish Oils Ltd., when that company was formed in the 1919 merger. He became general manager and a director in 1928 and in 1941 assumed the duties of managing director. He has taken an active part in industry affairs in many connections, but has been best known perhaps for his long and excellent services to the Institute of Petroleum, of which he is a Fellow. He served the branch in Scotland as chairman from its inception until his retirement last year from that office. Mr. Crichton is chairman of the West Lothian (Bangour) Hospital Group and is keenly interested in hospital work and in many aspects of local goverment. He has been a member of the West Lothian County Council for 30 years and its chairman for 10 years—a remarkable record of public service over a particularly difficu't period.

When he retires in March Mr. Crichton will have completed nearly 58 years' service with the Scottish shale oil industry.

DR. FELIX SINGER, the well-known ceramic consultant and author, left for Yugoslavia on 27 December, to act as adviser on the further development and application of research relating to clay and silicates at the University of Belgrade. Dr. Singer has gone at the request of UNESCO and will be away for three months. He is no stranger to Yugoslavia as he spent a short time there in 1951, following a request from the Yugoslavian Ministry of Metals.

Two additional directors have been appointed by Newman Industries Ltd.: MR. CHARLES BUSH, F.C.I.S., who has been company secretary since 1950 and will continue in that position, and MR. GEORGE REDFERN, M.A. (Cantab), A.M.I.E.E., who has been chief engineer of the company since 1950 and will continue as such.

MR. NATHANIEL ALLAN, a director and works manager of Fullerton, Hodgart & Barclay Ltd., Paisley, has been presented with an inscribed gold watch to mark his completion of 60 years' service with the company.

MR. COLIN OFFICER, of Edinburgh, retired at the end of 1953 after 52 years' service with J. F. MacFarlan & Co. Ltd., the Edinburgh manufacturing chemists. During much of this period Mr. Officer acted as an outside representative of the company.

The Canadian Section of the Society of Chemical Industry has announced that the Society of Chemical Industry (Canada) Medal for 1954 has been awarded to DR. R. K. STRATFORD, of Imperial Oil Ltd., Sarnia, Ontario. The medal will be presented at a meeting in Toronto on 24 February, when Dr. Stratford will deliver his address on 'The Scientist—His Place in Modern Society.' The award—given biennially—is in recognition of 'outstanding industrial or scientific achievement in connection with the Canadian chemical industry.'

Obituary

The death has occurred of MR. FITZROY KEITH CHAPMAN, chairman of Spencer Chapman & Messel Ltd. Mr. Chapman, who was 82, had been ill several weeks, but occupied the chair at the annual meeting of the company last October and continued to take an active interest in the business until within a few weeks of his death. He was the eldest son of the late Mr. Spencer Chapman, who founded the firm about 80 years ago. It became a company in 1899 and Mr. F. K. Chapman was appointed a director in 1905, taking over the chairmanship on his father's death in 1918. He was much esteemed by the staff, many of whom had been with the company for long periods.

We regret to record the death of DR. FREDERICK NEURATH, who for several years had been a regular contributor to THE CHEMICAL AGE. He had been very ill in hospital for some weeks and then seemed better, but had a relapse, followed by pneumonia. At one time Dr. Neurath was a manager of the Blackley Chemical Works Levinstein Ltd., now part of the I.C.I. organisation, and later held a Ministerial post in Vienna. He came to England in 1939, later becoming a British subject. In 1948 he was awarded the Golden Doctor Diploma of the University of Heidelberg, where 50 years previously he became a Doctor *Philosophiæ Naturalis*. His doctor's dissertation dealt with the synthesis of camphor-like compounds and was honoured with the distinction *cum laude superavit*. In his Golden Doctor Diploma he was hailed as a scientist who, in responsible positions in the chemical industry, had always played an outstanding part in its development.

Production Started

A $3,500,000 caustic-chlorine plant built by Western Chemicals Ltd., has started production at Duvernay, on the North Saskatchewan river about 70 miles each of Edmonton. Natural gas from the company's own wells will supply the plant with fuel, and salt, the main raw material, will come from other company-owned property. Present daily capacity is 10 tons of chlorine, 12 tons of caustic soda and 10 tons of muriatic acid. This will be expanded later.

German Oil Merger

A merger has been agreed to by Ebano-Raffinerie AG, Hamburg, and Esso AG, German subsidiary of the Standard Oil Co., Esso having acquired all the assets and liabilities of Ebano. The Ebano refinery has been processing crude oil for Esso as an independent enterprise.

Publications & Announcements

THE largest manufacturers of power spraying machines in Europe, Pest Control of Cambridge, report having designed and commenced production of a spraying machine exclusive to The International Harvester Company of Great Britain under the trade name of 'Weedall.' A high and low volume sprayer for Harry Ferguson Ltd., this is the second machine produced by this company for Harry Ferguson Ltd. in 12 months. In the last few days a contract has been signed with David Brown Ltd. for a low volume sprayer to be marketed in association with that company. A low volume sprayer has also been designed by Pest Control Ltd. for use with the Fordson Major.

* * *

APPLICATIONS Brochure No. 6, 'The Use of Aluminium Alloys in Structural Engineering', was published by the Aluminium Development Association in 1950 as an introductory survey to the subject, designed to make known the characteristics and uses of aluminium to a fairly wide audience. An important characteristic of aluminium in the field of structural engineering is the facility with which structural sections can be produced by the extrusion process and the fact that such sections allow freedom for disposing metal as required with consequent economies in production. A range of standard sections was published in BS.1161, 'Aluminium and Aluminium Alloy Sections'. An addendum to Brochure No. 6 has now been published, as a result of recent researches and the development of a range of new shapes to overcome certain problems inherent in thin sections. The data presented on some bulbed and lipped sections are shown in similar form to the existing data in BS.1161. Three sets of data are shown, covering equal bulb angles, unequal bulb angles and lipped channel sections. The publication concludes with some brief notes on the design data, including a comparison between the recommended strut curve for a channel section with the usual Perry curve for the same material

No. 23 of the Association's 'Aluminium Courier' is concerned with 'Cycles, Motor Cycles, etc.', and copies may be obtained, price 1s., from 33 Grosvenor Street, W.1.

THE second issue of 'Glaxo Topics,' the new bulletin for retail chemists issued by Glaxo Ltd., of Greenford, Middlesex, contains a report of trials on pencillin administration by mouth. The advantage of oral pencillin is that it may be given at frequent intervals without disturbing the patient's routine, and without the use of a syringe.

* * *

IMPREGNATION of packaging materials with pyrethrum and its synergists is finding favour in the prevention of damage by insects to foodstuffs in storage, according to S. V. Pingale and N. S. Kapur, of the Central Food Technological Research Institute, Mysore, in the course of an article in the July issue of 'Pyrethrum Post' (official publication of the African Pyrethrum Research Council), which has just reached us. The authors state that both chemical and biological methods were attempted to determine the differences in absorption of pyrethrins by jute, but they discuss only the results obtained by chemical analysis. Other articles in this issue include 'Use of Pyrethrum in Malaria Control' and 'Chromatographic Separation of the Pyrethrins.' Copies of 'Pyrethrum Post' are obtainable from the publishers at Anglo-French House, 2 Queen Anne's Gate, London, S.W.1.

* * *

SHEEPBRIDGE Engineering Limited have announced that by agreement with the United States Pipe and Foundry Co., Special Products Division, Burlington, New Jersey, the special method developed by the American firm for making long cast cylinders and tubes can be employed by two Sheepbridge subsidiaries—Sheepbridge Steel Castings Ltd., Sutton-in-Ashfield, Notts, and Sheepbridge Stokes Ltd., of Chesterfield. A prominent feature of this new horizontal rotation process is the use of a special refractory spray round the walls of the mould that induces the casting metal to flow evenly throughout its length, thereby eliminating a principal difficulty in normal British practice. The intention of Sheepbridge Steel Castings, Ltd. to spin cast piping in high alloy steels and nickel base alloys by this process has aroused considerable interest in the chemical industry, where long piping in special alloys is difficult to produce by normal methods.

INDUSTRY has been interested for more than 30 years in the possibilities of using natural rubber latex in the manufacture of paper and analogous products and this is the subject of one of the articles in the winter edition of 'Rubber Developments,' published by the British Rubber Development Board, Market Buildings, Mark Lane, London, E.C.3. Among other articles of particular interest is one dealing with the use of rubber in the laboratory.

* * *

DUE, no doubt, to the satisfactory results obtained in the gas turbine field from the use of Nimonic 75 for combustion chamber linings, Nimonic alloys are becoming well established in many fields where there is need for a high-temperature oxidation-resistant material. The two alloys of the Nimonic series which are most widely used in sheet form are Nimonic 75 and its modified form, Nimonic F. A new publication, issued by the manufacturers, Henry Wiggin & Co. Ltd., Wiggin Street, Birmingham 16, gives up-to-date information on the annealing and pickling of these alloys, replacing earlier fabricating instructions. This includes notes on temperatures for annealing and furnace atmospheres, together with formulæ of acid pickles and alkaline liquors, and details of salt bath descaling. Copies may be obtained from the company.

* * *

'WIGGIN Nickel Alloys' No. 23 reviews work carried out on crop drying by electricity, a major development in farming. Types of industrial heating elements, ultra-violet and infra-red ray radiation, filtration, incubation, cathode ray tube seals, temperature control, electrical upsetting and the introduction of Nimoply are other subjects dealt with. Copies of this journal may be obtained free of charge by writing to Henry Wiggin & Co. Ltd., Thames House, Millbank, London, S.W.1.

* * *

NEW reagents available from BDH include diethyl-ammonium diethyl-dithiocarbamate, for the separation of small amounts of arsenic, copper and bismuth from lead and zinc; naphthidine for internal indication in the titration of zinc with ferryocyanide; and *iso*-nitroso-malonyl guanidine ammonium salt, which will detect cobalt as low as 1 part in 20,000,000. Other additions are 1, 3-dimethyl-butylamine, and improved reagents for the BP arsenic test.

COURLOSE — the registered trade mark applied by Courtaulds Ltd., 16 St. Martin's-le-Grand, London, E.C.1, to their sodium carboxymethyl cellulose — is the title and subject of a booklet just issued by that company. Respective chapters deal with the properties of Courlose, its properties, the grades in which it is available, its uses and the preparation of solutions. There is also a select bibliography, which is most useful.

* * *

LATEST development by W. G. Bagnall Ltd., locomotive builders, of Stafford, are diesel mechanical shunters of the 040 and 060 type designed primarily for industrial shunting in steel works, factories, mines, harbours and on plantations. These locomotives are powered by McLaren 'M' series, vertical, direct injection, cold-starting diesel engines. The engine drives through a Vulcan-Sinclair fluid traction coupling and a flexible coupling of the Layrub type to a constant mesh gearbox giving four speeds. The changes are effected by four oil-operated cone-type couplings. The reversing mechanism consists of bevel gears and reversing is effected by means of a simple dog clutch. The final drive is from a jackshaft with fly cranks connected to the locomotive wheels by coupling rods.

* * *

THE TUV Germicidal Tube manufactured by Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2, which is already in extensive use in many branches of the food industry, is now being applied to long-standing problems in the soft drinks trade. Many of the sugars, syrups and fruit juices used in the manufacture of soft drinks tend to deteriorate in storage, the action of micro-organisms giving rise to destructive fermentation and scum. Similar effects are apparent with flavouring extracts and edible oils. The condensation of moisture on the tops and sides of the storage tanks dilutes the top layer sufficiently to allow micro-organic growth and spores may float up from within the liquid to the air, there to germinate. To combat this problem several well-known firms, including Schweppes Ltd., L. Rose & Co. Ltd. and Idris Ltd., have installed Philips TUV tubes. These tubes are fitted in an easily accessible position as near to the top of the tank as possible, where constant irradiation ensures the sterility of the air space above the liquids. Tests have proved the efficacy of this method.

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The most reliable method of affixing cartridge paper-lined tin tagger capsules, thus ensuring a perfectly fitting closure for sealing tins and drums containing liquids.

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Law & Company News

Commercial Intelligence

The following are taken from the printed reports, but we cannot be responsible for errors that may occur.

Mortgages & Charges

(Note.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described herein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every company shall, in making its Annual Summary, specify the total amount of debt due from the company in respect of all Mortgages or Charges. The following Mortgages or Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an *—followed by the date of the Summary but such total may have been reduced.)

INDUSTRIAL & SCIENTIFIC INSTRUMENTS LTD., London, W.C.—26 Nov., 1953, £1,000 bill of sale, to R. Lee, London; charged on specified machines, tools, etc. *Nil. 2 January, 1951.

LEDA CHEMICALS LTD., London, W.—19 Nov., 1953, mortgage to Midland Bank Ltd., securing all moneys due or to become due to the bank; charged on part of factory premises at Wharf Road, Ponders End, with fixtures. *£6,132. 22 December 1952.

METALLO CHEMICAL TRUST CO., LTD., London, E.C.—5 Nov., 1953, agreement securing to Industrial and Commercial Finance Corporation, Ltd. £40,000 charged on certain debenture stock. *Nil. 7 October, 1953.

Satisfaction

KAY'S (RAMSBOTTOM) LTD., soap and chemical manufacturers.—Satisfaction, 28 Nov., 1953, debenture registered 8 Nov., 1952, to the extent of £5,000.

New Registrations

H. J. Tuff Ltd.

Private company. (527,004.) Capital £100. Pharmacists, chemists and drug stores, manufacturing chemists, manufacturers of cosmetics. Directors: Harry J. Tuff and Mary B. Tuff. Reg. office: 2a Harmer Street, Gravesend, Kent.

Derek Adams & Co. (Bridgend) Ltd.

Private company. (527,044.) Capital £5,000. Manufacturing chemists and pharmaceutical chemists, etc. Directors: Derek Adams, Miss Dorothy M. Burnard, Miss Lucy Adams, Harry Hislop and Ronald B. A. Toms. Reg. office: Offices of Graham Paul & Co., Queens Road Trading Estate, Bridgend.

P.E.D. Medicinal Preparations Ltd.

Private company. (527,009.) Capital £100. Objects: To enter into an agreement with Medicinal Chemicals Ltd.; to carry on the business of research and manufacturing chemists, importers, exporters and manufacturers of and dealers in pharmaceutical, medicinal, chemical, industrial and other preparations, etc. First directors to be appointed by the subscribers. Reg. office: 36 Victoria Street, London, S.W.1.

Delalande Ltd.

Private company. (527,361). Capital £1,500. Manufacturers of and dealers in pharmaceutical preparations, chemicals, gases, drugs, medicines, etc. Directors: Fredk. G. Maw and Michel L. Delalande. Reg. office: Stafford House, Norfolk Street, Strand, London, W.C.2.

General Detergents Ltd.

Private company. (527,368). Capital £1,000. Industrial and general chemists, druggists, etc. First directors not named. Reg. office: 1 Metal Exchange Buildings, Whittington Avenue, London, E.C.3.

Reckitt & Colman Holdings Ltd.

Private company. (527,217). Capital £2. Objects: To acquire not less than 90 per cent of the issued share capitals of Reckitt and Sons, Ltd., and J. and J. Colman Ltd. Power is taken to carry on business as chemists, druggists, drysalters, oil and colour men, starch manufacturers, etc. First directors not named. Solicitors: Slaughter and May, 18 Austin Friars, London, E.C.2.

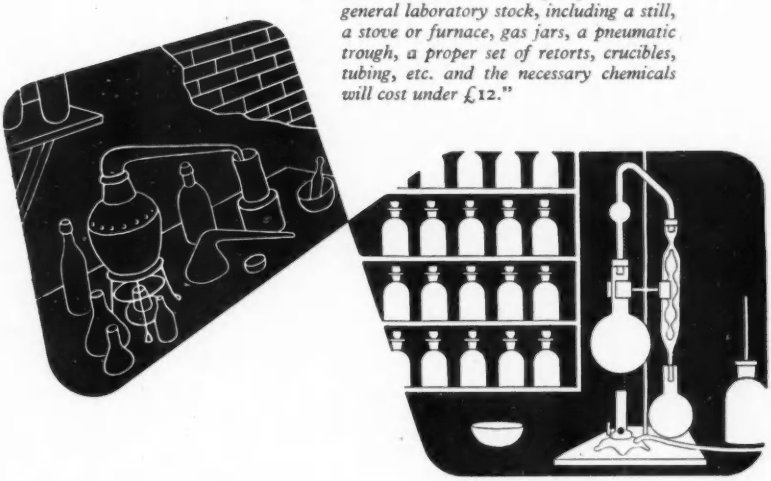
Maniplastics Ltd.

Private company. (527,154). Capital £100. Manufacturers, importers and exporters of and dealers in bakelite, celluloid, cellulose, acetates and ebonites and other substances; designers and manufacturers of machinery and machine tools for the moulding and extrusion of all such plastics, chemicals and allied substances, etc. Directors: Henry E. E. E. Barton, Christopher G. Colclough, Peter J. Garrini and John R.

[continued on page 192

A rough and shabby room

"For teaching chemistry, a laboratory is absolutely essential No matter how rough and shabby a room, so that it be well ventilated, have gas and water laid on, and will hold sixteen to twenty boys . . . . the general laboratory stock, including a still, a stove or furnace, gas jars, a pneumatic trough, a proper set of retorts, crucibles, tubing, etc. and the necessary chemicals will cost under £12."



Standards, like prices, have gone up since 1869, when the first issue of Nature reproduced an address presented by the Rev. W. Tuckwell to the British Association Meeting in Exeter containing this passage.

Much good work was done in the "rough and shabby rooms" of a century ago; but the layout and equipment of the modern laboratory, and the stocks of B.D.H. reagents on its shelves, bear witness to the standards that are considered essential to-day.

LABORATORY **B·D·H** CHEMICALS

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Company News

British Xylonite Co. Ltd.

The selling activities of the Halex Division of the British Xylonite Co. Ltd.—hitherto carried out through a subsidiary company, Halex Ltd.—are now being carried out by the division itself under the style of 'Halex —a division of the British Xylonite Co.'

Reckitt & Sons and J. & J. Colman

A statement issued jointly by Reckitt & Sons Ltd. and J. & J. Colman says that for some time past the two boards have had under consideration the desirability of completing the amalgamation of the companies, whose trading activities have since January, 1938, been merged in the jointly owned company, Reckitt & Colman. The boards have agreed that the time has now arrived for completing the amalgamation in the interests of the stockholders of both companies. Steps have been taken to form a new holding company with the name Reckitt & Colman Holdings, which will offer to acquire the issued preference and ordinary stocks of Reckitt & Sons, Ltd., and of J. & J. Colman.

Union Carbide Canada Ltd.

Union Carbide Canada Ltd. has been formed as a subsidiary of Union Carbide and Carbon Ltd. of the United States to co-ordinate the operations of six Canadian companies which have been operated as units of the US parent firm—Bakelite Company, Canadian Railroad Service Co., Carbide Chemicals Sales Co., Dominion Oxygen Co., Electro Metallurgical Co., and National Carbon Co. These are now divisions of the Canadian company.

Erinoid Ltd.

In a review circulated in connection with the recent annual meeting of Erinoid Ltd., the chairman, Mr. C. E. Cleeve, stated that for the last financial year the company had a trading profit of £87,607 and after providing £51,520 for taxation there was a net profit of £36,087. The revival of demand for casein had been maintained, but the demand for cellulose acetate moulding powder had again been disappointing. The final ordinary dividend was 5 per cent.

Monsanto Plastics Ltd.

Montanto Chemicals Ltd. announce the formation of a new and wholly owned subsidiary company, Monsanto Plastics Ltd., registered on 31 December, 1953, with an initial capital of £100. Directors of the new company are J. W. Barrett, D. R. Mackie, N. F. Patterson, E. L. Pixton, P. A. Singleton, and J. W. Urban. The secretary is R. E. H. Sheridan, assistant secretary of Monsanto Chemicals Ltd. The operating address of the new company is Victoria Station House, Victoria Street, London, S.W.1. Monsanto Plastics Ltd. has been formed for the purpose of consolidating and extending Monsanto's activities in the plastics field.

Canadian Paper-Alcohol Merger

The directors of Canadian International Paper Co. recently voted to make offers to the shareholders of Commercial Alcohols Ltd., to purchase their shares at $4 for each common share and $6.50 for each preferred share. Later, the directors of Commercial Alcohols Ltd. unanimously agreed to recommend to their shareholders acceptance of the offers made by Canadian International Paper Co. The plant of Commercial Alcohols Ltd. at Gatineau, Quebec, opened in 1949, produces all the basic alcohols used in the firm's various products from sulphite waste liquor obtained from the adjacent plant of Canadian International Paper. It would appear to be the view of CIP that the alcohol plant could function more efficiently and economically as an integrated operation making it an auxiliary unit of CIP. Commercial Alcohols manufactures pure and denatured ethyl alcohol, radiator anti-freeze, a gasoline conditioner, magnesia insulating materials, and shellacs.

Columbite-Tantalite Imports Up

Imports of columbite and tantalite concentrates, from which the strategic metals columbium and tantalum are produced, were half as large again during the first nine months of 1953 as during all of 1952. USA Secretary of the Interior, Douglas McKay, announced recently. The United States depends upon foreign sources for about 99 per cent of its requirements for columbium and tantalum. During 1952, for instance, domestic mine shipments totalled 5,385 lb.; imports exceeded 2,000,000 lb.

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ZEO-KARB 216	A resin containing weak acid groups of the carboxyl type.	**BIO-DEMINROLIT**	A mixed cation and anion exchange resin of high stability for demineralisation in a single column.
ZEO-KARB 315	A sulphonic acid resin particularly stable up to 100°C.	**DECOLORITE**	A resin of high porosity for removing colour from solutions.
ZEO-KARB 225	A unifunctional, high capacity sulphonic resin in bead form.	**RESIN MEMBRANES**	For special purposes, many of these materials can be supplied as membranes in the form of rods, discs and thimbles.

With forty years' experience in the manufacture and operation of Ion Exchange materials, the Permutit organisation is continually developing new materials, and new methods of using them. Its Research Laboratory is ready always to co-operate in the solution of your problems.

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Next Week's Events

MONDAY 11 JANUARY

Society of Chemical Industry

Leeds: The University (Chemistry Lecture Theatre), 7 p.m. Joint meeting of Yorkshire Section with Oils & Fats Group. G. C. Hampson: 'Synthetic Detergents.'

Institute of Fuel

Newcastle-on-Tyne: King's College (Chemistry Lecture Theatre), 6.30 p.m. J. Brown: 'The Calorific Value of Coal.'

TUESDAY 12 JANUARY

Royal Institute of Chemistry

Gravesend: Technical College (Mayfield Hall Annexe), Pelham Road, 7.30 p.m. Joint meeting with Gravesend & District Engineering Society. C. A. Robiette: 'Corrosion & the Schori Spraying Process.'

Chemical Society

Exeter: Washington Singer Laboratories, 5 p.m. Joint meeting with RIC. Dr. A. G. Maddock: 'The Chemical Effects of Nuclear Transformations.'

Society of Chemical Industry

London: Geological Society's apartments, Burlington House, Piccadilly, 5.30 p.m. Chemical Engineering Group meeting. F. E. Warner, A. M. Cook and D. Train: 'Engineering & Microbiological Processes.'

Belfast: Agriculture Lecture Theatre, Elmwood Road, 7.15 p.m. E. M. Lindsay: 'Recent Advances in our Knowledge of the Universe.'

Institute of Fuel

London: Waldorf Hotel, Aldwych, 5.30 p.m. Joint meeting with Coke Oven Managers' Association (Southern Branch). D. T. Barrit and T. Kennaway: 'New Carbonisation Processes Under Development & Their Relation to Established Practice.'

Institute of Metals

Swansea: University College (Metallurgy Department), Singleton Park, 6.45 p.m. J. Crowther: 'Continuous Casting.'

Royal Statistical Society

Newcastle-on-Tyne: Crown Hotel, Clayton Street, 7 p.m. M. J. Moroney: 'Machine Utilisation.'

WEDNESDAY 13 JANUARY

Society of Chemical Industry

London: Institution of Civil Engineers, Great George Street, Westminster, 2.15 p.m. Joint meeting of Microbiology Group with Society for Applied Microbiology. Symposium: 'Industrial Methods of Sterilisation.'

THURSDAY 14 JANUARY

Chemical Society

Bristol: The University (Department of Chemistry), 7 p.m. Joint meeting with RIC and SCI (Food Group and Bristol Section). R. V. Wadsworth: 'Cacao Diseases.' (Preceded by afternoon visit to factory of J. S. Fry & Sons, Ltd., Somerdale).

Edinburgh: North British Station Hotel, 7.30 p.m. Joint meeting with RIC and SCI. Professor D. H. R. Barton: 'The Stereochemistry of *cyclo*Hexane Derivatives.'

Society of Instrument Technology Ltd.

London: Manson House, Portland Place, W.1, 6 p.m. Control Section annual general meeting followed by H. Williamson: 'The Theory & Design of Compound Action Automatic Controllers with Particular Reference to the Interaction Factor.'

FRIDAY 15 JANUARY

Royal Institute of Chemistry

St. Andrews: United College (Chemistry Department), 5.15 p.m. Joint meeting with CS. Dr. F. H. Pollard: 'Recent Advances in Inorganic Chromatography.'

Chemical Society

Newcastle-on-Tyne: King's College (Chemistry Building), 5.30 p.m. Bedson Club Lecture by Professor E. Baldwin.

Swansea: University College, 5.30 p.m. Joint meeting with University College of Swansea Chemical Society. Professor M. J. S. Dewar: 'Some Recent Developments in Theoretical Organic Chemistry.'

Society of Chemical Industry

London: King's College, Strand, 4.30 p.m. Fine Chemicals Group meeting. Dr. Tudor S. G. Jones: 'Paper Chromatography & Electrophoresis' (discussion and demonstrations).

Glasgow: Royal Technical College, 7.15 p.m. Joint meeting with RIC. Film display.

Society of Dyers & Colourists

Manchester: Textile Institute, 6.30 p.m. S. Blackburn and M. R. Fox: 'The Behaviour of Leuco Vat Dyes Above 100°.'

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A new edition of " Modern Dust Collection and Fume Removal " is now available. In this well illustrated booklet the important subjects of Dust Collection and Fume Removal are fully dealt with. Numerous examples are also given of effective methods of controlling airborne dust in various industries.

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Australian 'Catcracker'

CONSTRUCTION work on one of the first 'catcracker complexes' (catalytic cracking units) to be built in Australia is planned to begin early this year at Geelong Refinery, near Melbourne. Scheduled for completion about the middle of 1955, the new plant will have an intake capacity of 650,000 tons a year and will have the effect of converting heavy oil fractions to motor spirit, thus making an important contribution to Australia's requirements of that commodity.

The project forms part of a construction programme designed to make Geelong one of the largest and most modern oil refineries in the Commonwealth. The first stage in the programme—comprising a crude distillation unit with a capacity of approximately 1,750,000 tons a year and ancillary treating facilities—is expected to be completed in the spring.

The 'catcracker complex' will include a feed preparation unit to prepare the feedstock for the 'catcracker' proper and later a polymerisation plant for converting a portion of the valuable catcracked gases to high grade motor spirit. When this complex is completed, Geelong's tankage will consist of some 60 storage tanks with a capacity of approximately 400,000 tons.

Aspects of Biochemistry

DESIGNED primarily for chemists who require an insight into biochemical training, a course of 12 lectures on 'Some Aspects of Biochemistry,' has been arranged to be held at Acton Technical College, High Street, Acton, W.3, during the 1954 spring term.

The course will also be of value to those already engaged in biochemical work and who require to revise and refresh their knowledge in this field — in general it should appeal to those employed in production and control of chemicals of physiological or medical importance.

The lectures will be given by Dr. A. E. Bender, Ph.D., B.Sc. (Biochemistry), F.R.I.C., senior lecturer, The Crookes Laboratories Ltd., on Wednesdays beginning at 7.30 p.m. and further details follow:

13, 20, 27 *January and* 3 *February*: 'The Elements of Nutrition.'

10, 17, 24 *February and* 3 *March*: 'The Enzymes—Their Nature and Function.'

10, 17, 24, 31 *March*: 'Intermediary Metabolism.'

Early application is advised as accommodation is limited. The fee for the course is £1 10s.

Market Reports

LONDON.—The movement at the beginning of the year has been on a good scale with a broadening interest in most sections of the market. As is usual for the period there has been a fair weight of contract replacement buying by the consuming industries, and with a few exceptions existing rates are continued.

The price of oxalic acid was reduced by £11 10s. a ton, as from 1 January, the new quotation being £127 10s. per ton in 5-cwt. casks, carriage paid. The basis prices for red and white lead remain unaltered. While the home trade supplies are fairly good some items such as the barium salts are not readily available for export. Sodium fluoride is reported to be dearer on export account.

Most sections of the coal tar products market are on a firm basis with persistent inquiry for xylol and solvent naphtha.

MANCHESTER.—A fairly steady resumption of trading operations after the holidays has been reported on the Manchester market for chemical and allied products during the past week. Leading industrial consumers have in a good many instances covered their requirements over the next few months and deliveries under these contracts, especially to the textile and allied trades, are going forward satisfactorily. A fair aggregate weight of new business has been placed during the past few days. Creosote oil and most of the light tar products are also going steadily into consumption. In the market for fertiliser materials bookings during the week have been on a moderate scale and a gradual seasonal improvement in the demand for these is expected.

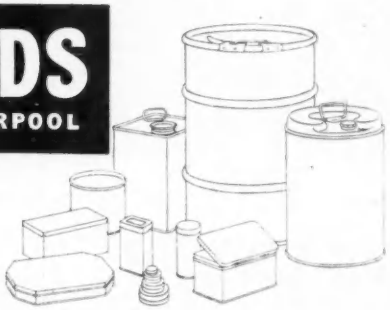
GLASGOW.—Apart from the usual interest at this time of year due to the renewal of contracts, etc., business has been quiet. However, this is quite normal at such a time and with the amount of business on hand it looks as though the New Year is opening with very good promise.

A GREAT RANGE

In Astronomy
it's Mount Palomar
of California
Scientific advances yet undreamed of are expected from the new 200 inch telescope at Mount Palomar, California. Largest of its kind in the world, this giant aid to interstellar exploration measures 55 feet in length, 20 feet in diameter and weighs 250 tons. No wonder it dwarfs members of the observatory staff who stand beside it!

in tins, cans and drums it's

READS
OF LIVERPOOL



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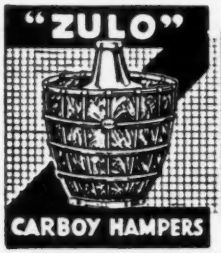

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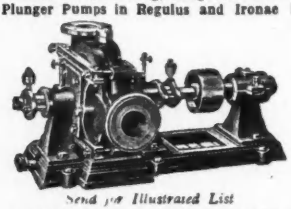

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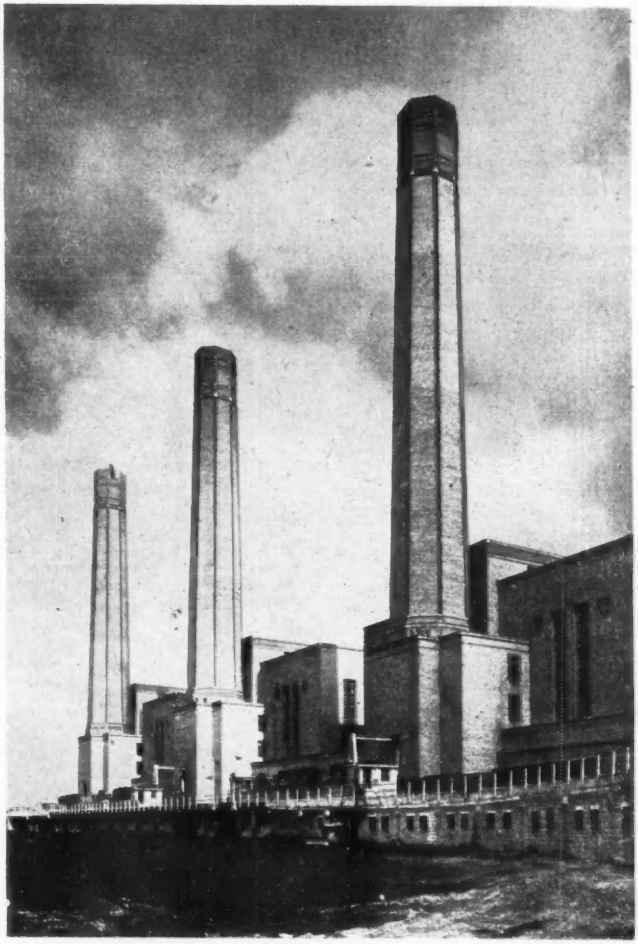

Three brick chimneys 300 ft. high built by us at Staythorpe Power Station, for the East Midlands Division of the British Electricity Authority. Main Contractors and Consultants—Messrs. Balfour Beatty & Co., Ltd. Divisional Controller—L. F. JEFFREY, Esq., B.Sc., M.I.C.E., M.I.Mech.E., M.I.E.E.

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CLASSIFIED ADVERTISEMENTS

SITUATION VACANT

The engagement of persons answering these advertisements must be made through a Local Office of the Ministry of Labour or a Scheduled Employment Agency if the applicant is a man aged 18-64 *inclusive, or a woman aged* 18-59 *inclusive, unless he or she, or the employment, is excepted from the provisions of the Notifications of Vacancies Order*, 1952.

VACANCIES FOR STAFF in Manchester factory, for the making of **BROMIDE PHOTOGRAPHIC EMULSIONS** and the operation of **BROMIDE COATING PLANT. BOX No. C.A. 3287, THE CHEMICAL AGE, 154, FLEET STREET, LONDON, E.C.4.**

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50 Tons Kentish Hardwood **CHARCOAL**, granulated, 1 in. to $^{1}/_{25}$ in., for sale in 5-ton lots, for case hardening or gas absorption. **BOX No. C.A. 3288, THE CHEMICAL AGE. 154, FLEET STREET, LONDON, E.C.4.**

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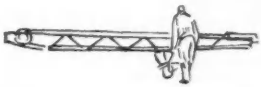

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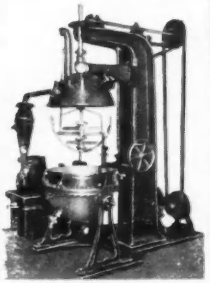

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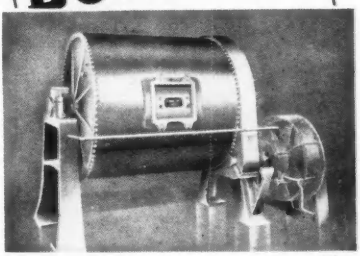

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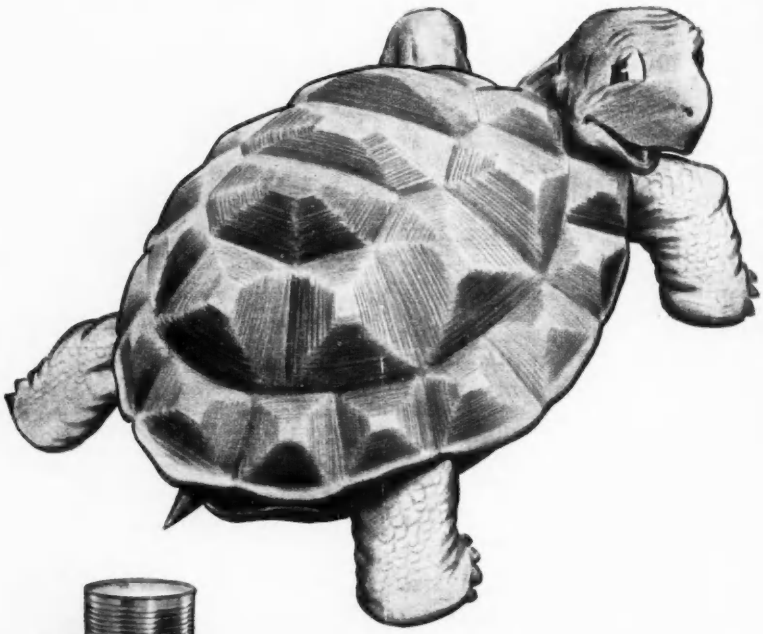

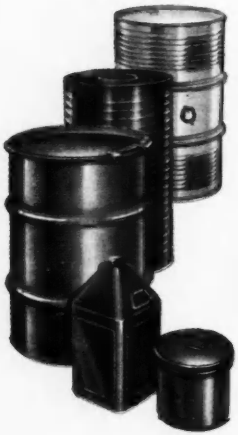

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